United States Court of Appeals

for the

District of Columbia Circuit



TRANSCRIPT OF RECORD

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Joseph W. Stewart

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BRIEF FOR APPELLANT

IN THE

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA

No. 8016

84

EZRA LEVIN, *Appellant*

v.

CONWAY P. COE, COMMISSIONER OF PATENTS

Appeal from the District Court of the United States for the District of Columbia.

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v.

CONWAY P. COE, COMMISSIONER OF PATENTS

Appeal from the District Court of the United States for the District of Columbia.

BRIEF FOR APPELLANT.

I. JURISDICTIONAL STATEMENT.

This Court has jurisdiction of the present appeal because it was duly taken from a final judgment of the District Court of the United States for the District of Columbia in a suit brought by Ezra Levin, appellant, under Section 4915 of the Revised Statutes (U. S. C. Title 35, Sec. 63) against defendant, a resident of the District of Columbia in his official capacity as the Commissioner of Patents.

II. STATEMENT OF THE CASE.

The judgment of the District Court dismissed plaintiff's complaint seeking a patent under Section 4915 of the Revised Statutes.

The invention in suit is defined in claims 3, 4, and 6 to 16 inclusive of application Serial No. 94,709, filed August 6, 1936. That application was a continuation-in-part of application Serial No. 683,085, filed July 31, 1933.

The substance of the claims relates to a method of making a palatable stable wheat germ product of high vitamin B1 content and for the product of that process.

The claims were all rejected by the patent examiner together with two others which were allowed by the Board of Appeals of the Patent Office.

Claim 15 to the method and claim 3 to the product are typical claims:

> 15. The method of treating grain germ to extract therefrom substantially all of the oil without affecting its vitamin content, which comprises extracting from grain germ substantially all of the oil with a heated solvent which has hydrolyzed to form an acid, removing the majority of the solvent by drainage, removing the last traces of solvent in the presence of heated moisture and under a vacuum at a temperature substantially below the boiling point of water, the grain being maintained in an acid condition during the entire extraction process.
>
> 3. A finished stable, marketable, non-toxic and palatable dry food product with a high content of vitamin B, consisting of all the constituents of natural grain germ except the oily constituents thereof and with such constituents unimpaired and in the same condition as in the natural germ.

The art cited by the Patent Office and relied upon in this appeal is:

Hoffman, 1,640,193.
Sure, 1,869,721.
Langfeldt, 1,925,489.
Sherman "Chemistry of Foods" 1920, pp. 349-350.
Sherman & Smith "The Vitamins" 1922, page 45.
Yaryan, 241,772.

Wheat is one of the oldest forms of human vegetable food. The wheat kernel [1] consists of three divisions: the germ,[2] which is the embryo of the wheat; the endosperm, which is the mass of starchy material substantially surrounding the germ; and the bran or hull enclosing both endosperm and germ.

In the distant past the whole wheat kernel was used for food and, when the wheat was ground into flour, the flour also contained all the elements of the wheat. The wheat germ, however, quickly becomes rancid due probably to the oil it contains.[2]

Therefore, when it became customary to mill flour in large quantities rather than locally for individual use, means were devised for removing wheat germ from flour and for about seventy-five years flour did not contain the vital essentials of wheat germ.

About twenty or twenty-five years ago it was discovered that the wheat germ contained large quantities of Vitamin B. In fact it was found that Vitamin B was present in larger proportions in wheat germ than in any other natural food. Wheat germ also contains extraordinarily large proportions of available phosphorus, manganese, iron, proteins, copper, and other vitamins in the B complex.[3]

At least twenty years ago efforts were made to develop wheat germ and a source of these materials, particularly

1. Physical Exhibit 3.
2. Physical Exhibit 4. Levin appendix p. 22, Record p. 31.
3. Levin, appendix p. 18, Record p. 26.

of Vitamin B. Without exception these efforts were commercial failures except in the case of applicant. The Ogilvie Company, largest millers in Canada tried for many years to produce a stable wheat germ product and were unable to do it. They now buy from applicant.[3a] Some hundreds of millions of pounds of wheat germ are removed each year from wheat in the United States and are fed to cattle or thrown away.[4] This material contains vitamins and minerals for which the public pays millions of dollars a year in other forms, frequently synthetic.[5] The oil in wheat germ is what causes it to become rancid and unfits it for use in flour or as a commercial food. The oil may be removed readily by solvent extraction at the expense of spoiling the remainder of the germ. That is, the wheat germ may be treated with a solvent, and the solvent drawn off with most of the oil. The oil is then recovered from the wheat germ and in itself is a valuable product. The difficulty came in trying to remove the solvent from the wheat germ to such an extent as to leave it palatable.[6]

The removal of the last portions of solvent from the residue is a matter which presented grave difficulties and was not solved prior to Levin's invention.[7]

The art cited by the defendant consists essentially of the Hoffman patent[8] which is relied upon only as showing wheat germ oil which has been defatted but from which the solvent has not been removed. The argument of defendant is that it involves no invention to remove the solvent from a defatted wheat material. The other references are relied upon to show that an acid environment was known to be favorable to the existence of the B1 complex.

3a. Rich, appendix p. 36-37, Record pp. 50-51.
4. Levin, appendix p. 18, Record p. 26.
5. Levin, appendix p. 19, Record p. 27.
6. Levin, appendix p. 22, Record p. 31.
7. Levin, appendix p. 24, Record p. 33.
8. Appendix p. 84.

The method employed by applicant to get rid of the last traces of solvent is a highly ingenious one. He was unable to remove the last traces of ordinary solvents from the wheat germ but he did find that under the proper conditions certain chlorinated hydrocarbons such as ethylene dichloride and carbon tetrachloride would undergo a chemical reaction of a wholly unexpected type by means of which the last traces of solvent, instead of being distilled off, would *hydrolyze*.[9] Hydrolysis is a chemical reaction in which the ethylene dichloride reacts with *water* to form a chloride and an alcohol.[9] Neither of the final products of this new composition is unpalatable. In order to succeed the removal of solvent cannot be carried out EXCEPT in an atmosphere of steam.[10]

The Hoffman patent mentioned carbon tetrachloride as a possible defatting solvent for wheat germ. He also mentioned benzene. In neither instance did he attempt to remove the solvent or to prepare a wheat germ product. He did not employ a steam atmosphere. He did not disclose conditions suitable for hydrolysis. He did not teach hydrolysis. Benzene will not hydrolyze to remove last traces of solvent. On the other hand, he subjected the material to high temperature and finally took an aqueous extract of low vitamin content from it.

9. Levin, appendix pp. 23-24, Record pp. 32-34.
10. Levin, appendix p. 24-5, Record pp. 34-5.

III. STATEMENT OF POINTS.

Plaintiff assigned the following errors in the record and proceedings in the above entitled cause:

The District Court of the United States for the District of Columbia was in error as follows:

1. In refusing to authorize and direct the Commissioner of Patents to issue to plaintiff a patent upon the Ezra Levin application Serial No. 94,709, filed August 6, 1936, entitled "Method of Treating Cereal Grain Germ." This application was a continuation-in-part of an earlier application filed by plaintiff on July 31, 1933, Serial No. 683,085.

2. In sustaining and adopting the decision of the Patent Office, holding that the Ezra Levin application did not contain patentable invention.

3. In holding that the Hoffman patent No. 1,431,525, showing a process of malting a wheat germ product from which oil has been removed by solvent extraction from the wheat germ, constitutes sufficient disclosure to justify the rejection of the Ezra Levin application.

4. In holding that there was no invention in taking Hoffman's intermediate product and removing the solvent so as to fit it for human consumption.

5. In holding that a process whereby a palatable, stable wheat germ product of high vitamin content is obtained from the residue of organic materials by means of extracting the last traces of solvent from this residue by the chemical action of hydrolysis, which reaction it is disclosed unexpectedly continues to completion instead of being reversible as normally expected, is not invention.

6. In holding that a simple, inexpensive and efficient method of making available for public consumption an es-

timated $80,000,000 worth of vitamin B complex (present wholesale valuation), did not involve invention.

7. In failing to hold that the solution of a problem such as the one presented here constitutes invention, when to the knowledge of those working in the field the problem has continually existed yet evaded solution by many workers over a period of years.

8. In entering the judgment of June 17, 1941, dismissing the complaint with costs against plaintiff and holding there was no invention.

9. In refusing to enter each and every of the plaintiff's "Findings of Facts" as submitted as follows:

1. The principal basis of the Board of Appeals decision was on a factual matter, as to which additional evidence has been presented here.

2. Wheat is one of the oldest forms of human vegetable food. The wheat kernel consists of three main divisions, the germ, the endosperm, or starch portion, and the bran or hull.

3. In former times the whole wheat kernel was used for food and when the wheat was ground into flour, the flour also contained all the elements.

4. The wheat germ, however, quickly became rancid after grinding, and when flour was ground in large quantities, the millers found it necessary to remove the germ, and did so some seventy-five years ago.

5. Some 20-25 years ago it was discovered that vitamin B, existed in wheat germ, and in fact existed there in larger proportions than in any then natural food. Wheat germ contains extraordinarily large proportions of available phosphorus, manganese, iron, proteins, copper and other vitamins of the B complex.

6. Efforts commenced at least twenty years ago to develop wheat germ as a source of these materials and particularly vitamin B. The efforts have been failures except as to applicant's.

7. Some hundreds of millions of pounds of wheat germ are removed each year from wheat in the United

States, and are fed to cattle or thrown away. This material contains vitamins and minerals for which the public pays millions of dollars a year in other forms, frequently synthetic. The proteins, minerals and vitamins in wheat germ, if purchased at the drug store in their presently available forms would cost hundreds of millions of dollars per year.

8. The wheat germ is caused to become rancid because of the presence of wheat germ oil. This oil may readily be removed by solvent extraction, which consists in adding an organic solvent such as benzol or hexane to ground wheat germ and then draining off most of the solvent. By repeating this action most of the oil may be removed. However, the removal of the last portions of the solvent from the residue is a matter which presented difficulties and was not solved prior to Levin's invention.

9. The same is true of other organic material from which oil is removed by solvent extraction, and which are heat sensitive. Millions of tons of oil are extracted annually in this way from other products, and prior to Levin's invention the fibrous residue was unfit for human food.

10. The removal of a volatile liquid solvent from a liquid is a relatively simple matter. In most, but not all cases, one liquid may be separated from another by distillation, although it is always at the expense of some of each liquid. That is one liquid cannot be removed from another without also removing some of the other. These matters are covered by well known physical laws.

11. The separation of a liquid from a solid, and particularly from a fibrous solid is not governed by known physical laws. The matter is complicated by adsorption and absorption phenomena. For example water, which boils at 212° F. cannot be entirely removed from an ordinary glass vessel at temperatures far above 212° F. In fact water traces can remain on such glass vessel at red heat.

12. It takes only a very minute trace of solvent to spoil a food for human consumption. The old exam-

ple of the spoiling of bread by kerosene vapors, or the spoiling of food in the ice box by immeasurably small traces of vapors from cabbages or other odoriferous materials show this well.

13. Levin solved the problem by using a solvent which had a chemical action by which the last traces of solvent were removed. There is no cited art showing the use of such a principle for the removal of last traces of solvent.

14. The principle employed is that known as hydrolysis. This means a reaction with water which splits up a small proportion of the solvent. The reaction is reversible, that is, a balance or equilibrium is established under normal circumstances so that no matter how small the amount of solvent present, only a small percentage of it will hydrolyze.

15. Levin discovered, however, that in wheat germ, this hydrolysis, instead of being reversible went to completion.

16. Levin used chlorinated paraffinic materials as solvents, and steam as an aid to distillation and these chlorinated materials form chlorides which become stabilized in the wheat germ. The solvents are removed to the last trace.

17. The Hoffman patent shows a process in which oil is removed by solvent extraction from wheat germ. No attempt is made to remove the solvent from the wheat germ. Instead the germ is given an extensive high temperature treatment, is malted, a water extract separated, and the fibrous residue discarded.

18. Hoffman may use a chlorinated solvent, but he did not teach the use of steam or any distillation step in connection with it. His preferred solvent was benzol, where no hydrolysis would have occurred.

IV. SUMMARY OF ARGUMENT.

A.

The decision of the Board of Appeals of the Patent Office refusing a patent was based on the premise that the removal of the last traces of solvent from a fibrous food material was a simple matter and an expected thing. The uncontradicted evidence presented before the District Court (which was not before the Board of Appeals of the Patent Office) showed that there was no known instance of the removal of last traces of solvent from a fibrous, solid food product except applicant's. The removal of last traces of liquid from a solid does not follow known physical laws. This evidence required in and of itself a reversal of the decision of the Board of Appeals by the District Court; and the failure of the District Court to enter findings of fact in accordance with the uncontradicted evidence was obvious error.

B.

The evidence shows no teaching by anyone of the use of a hydrolysis reaction to remove last traces of solvent from any product. This is an entirely new reaction. From a theoretical point of view such hydrolysis should not act to remove the last traces of solvent.

C.

The testimony proves that the value of wheat germ was long known and for years a way was sought to prepare a stable, palatable wheat germ product for the market. Applicant was the first to solve this problem after himself working on it for many years and the product has met with commercial success. These facts by themselves entitle Levin to a patent.

Smith v. *Snow,* 294 U. S. 1 (79 L. ed. 721).

Paramount v. *American Tri-Ergon Corp.,* 294 U. S. 464.

D.

The findings of fact submitted by plaintiff were based directly, and almost word for word, upon the uncontradicted testimony. These findings should have been entered by the District Court.

V. ARGUMENT.

In the Levin process wheat germ is treated with ethylene dichloride in an extractor which consists of a removable foraminous basket. Solvent is flowed over the wheat germ and, when it covers it, is heated to a temperature of about 170° F. The oil dissolves in the solvent and overflows through a line 19 as shown in the drawings. Solvent is fed in the bottom of the extractor and passes out of the top with the dissolved oil.[1]

When the oil has been extracted, the solvent is drawn off so far as possible and then steam is supplied to vaporize the remaining solvent. In the presence of the steam and under the temperatures used, the ethylene dichloride hydrolyzes in part to form hydrochloric acid and ethylene glycol. This produces an acid environment which is beneficial to the retention of the vitamin B content of the wheat germ.[2]

After treatment the germ is light, dry, of a floury consistency, and quite palatable. There is no trace of taste of the solvent.[3] The resulting product has a vitamin B content actually higher than that of the original wheat germ.[4] This is due to the removal of the oil which contains very little wheat germ; so that on a proportional basis the vitamin B content of the remaining wheat germ is higher than that of the original germ.

The wheat germ also contains large amounts of minerals and other vitamins besides vitamin B1. It is extremely valuable as a food product.

The mineral and vitamin content of wheat germ were

1. Appendix pp. 41-42, Record pp. 180-182.
2. Appendix, pp. 42-43, Record pp. 181-183.
3. Appendix pp. 43-44, Record pp. 183-4.
4. Appendix p. 25, Record p. 35.

well known for years before Levin's development, but, in spite of this fact, millions of pounds of wheat germ were thrown away each year in the United States because of the fact that if left in the flour, the wheat germ caused it to become rancid. The vitamin and mineral content thus destroyed would have cost many millions of dollars a year in the best available substitute form.[5]

The rancidity of the wheat germ was due to the presence of oil. This was known and it was also known that the oil could be removed by extraction.[6] However, no one prior to Levin succeeded in extracting the material while leaving available the rest of the germ.[7] This was because the last traces of solvent could not be removed from the germ by any known process.[7] In fact there is no known fibrous food material from which the last traces of solvent have yet been extracted, except by applicant's process.[8]

The art cited by the Patent Office seems to us quite irrelevant. The two Sherman articles merely show that the removal of oil from wheat germ destroyed part of the toxicity of wheat germ. The Sure patent[9] is cited merely to show that vitamin B under certain conditions is more stable in an acid environment.

The Yaryan patent[10] (which was not cited in the Patent Office) merely shows the treatment of flax seed with a solvent and the extraction of some of the solvent from the material. There is no statement that all of the solvent is removed or that the resulting product is edible.

That reduces the prior art to the Hoffman patent.[11] Hoffman discloses a process by which wheat germ is first

5. Levin, appendix pp. 18-20, Record pp. 26-29.
6. See Hoffman, 1640193, appendix, p. 84.
7. Rich, appendix p. 37, Record p. 50.
   Levin, appendix, p. 24, Record p. 33.
8. Wurster, appendix p. 33, Record p. 46.
9. Appendix, p. 90.
10. Appendix, p. 95.
11. Appendix p. 84.

treated with a solvent such as benzene or carbon tetrachloride to remove the oil. After that the wheat germ is cooked under high pressure, cooled, acidified, and then malted. The liquor is then filtered and the filtrate evaporated to dryness. This dried filtrate is a non-fibrous crystalline material containing only a minor proportion of the available B1 and practically none of the other available materials of the germ. All of the fibrous material is left behind and *not* used.

It is not this final product which is cited by the Office but the intermediate oil-free solvent-containing material. The decision of the Patent Office was based on the premise that there was no difficulty in removing the solvent from this intermediate product.[11a]

As we have already pointed out, the Hoffman intermediate product is no better than the other prior art or applicant's admission. It was known that the oil could be removed from wheat germ by a solvent. This knowledge of course presumes that after the removal there would be solvent on the wheat germ. Hoffman adds nothing to this knowledge. Instead, he confirms our proof that the removal of the solvent was a matter of extreme difficulty. If the removal of solvent was a simple matter, why was it necessary for Hoffman to go through his long treatment and finally wind up with an aqueous extract containing only a small portion of the vitamin B1 of the original grain?

In this day of chemical advances and improvements of all kinds, it is perhaps hard for any individual to understand why solvent extraction from a fibrous material should be difficult. The proof shows that it is.[12] But while it is generally easy to separate one liquid from another, and while most of the solvent can generally be separated from

11a. Appendix p. 78.

12. Wurster, appendix p. 33, Record, pp. 45-46.

any solvent, the removal of the last tastable traces of a solvent from a fibrous material is generally impossible at temperatures which the material will stand. This is because our foods, like wheat germ, are unbelievably complicated. The removal of liquid does not proceed in accordance with the physical rules of distillation which apply to liquid separation.[13] All sorts of other factors become effective, such as adsorption, absorption, chemical reactions, and the like. It is even extremely difficult to remove last traces of water from glass. To do so requires great heat.[14] A food product cannot be heated to such temperatures.

The removal of last traces of a solvent from wheat germ is no less complicated than would be the removal of the last traces of alcohol from the human body. One cannot just turn on the heat. Our expert witness, Mr. Wurster, is one of the leading experts in the country on distillation and his firm of Wurster & Sanger is well known for the excellence of their commercial installations. Mr. Wurster is not a theoretical expert but is a practicing engineer. He stated that he knew of no other instance than that of Levin where solvent had been completely extracted from fibrous materials which were later to be used directly for human food.[15]

That testimony is uncontradicted and *must* control. The District Court disregarded it.

Besides Mr. Wurster's uncontradicted evidence that the removal of the last traces of solvent from foods is extremely difficult, common sense shows this to be so. We all know that many food products, such as bread and eggs, rapidly pick up odors from other substances. Those things which cause the odors are similar to the odoriferous materials which are used as volatile solvents. We probably

13. Wurster, appendix p. 33, Record p. 46.
14. Wurster, appendix p. 34, Record pp. 46-7.
15. Wurster, appendix pp. 33, 35, Record pp. 46, 48.

could not measure the amount of essence of onion which condenses in an egg, but any of us can taste it.

In the old days when we used to have to buy kerosene to run the kitchen stove, we were always instructed to keep the kerosene and the bread and eggs separate. The infinitesimal quantities of kerosene in the air would contaminate the bread and there was no way of getting rid of the taste.

That is why wheat germ could not be freed from the last traces of solvent prior to applicant's process. Applicant worked for many years before he was successful in making a palatable product and he did it only by making use of a hydrolyzable solvent. No previous use of a hydrolyzable solvent, under conditions where the last traces of it would hydrolyze, has been shown.

Hoffman *does* suggest the use of carbon tetrachloride which is one of the solvents which will work here, but he does not suggest removing it, and it is impossible to infer from him a suggestion of the conditions under which hydrolysis would occur to result in the removal of the last traces. On the contrary, Hoffman teaches that benzene, which will not hydrolyze, is equivalent to carbon tetrachloride which will. Hoffman does not teach hydrolysis, use hydrolysis, or show conditions under which it would be effective to remove last traces of solvent. He does not suggest the use of steam distillation, which is essential.

As a matter of fact the hydrolysis reaction is not one which would be expected. It is what is known as a reversible reaction in chemistry. This means that there should always be a balance between certain amounts of material on each side of the reaction. The reaction of ethylene dichloride with water to form hydrochloric acid and a glycol should never go to completion, unless in some way we remove one of the products to unsettle the balance. That is, there should always be some of all four constitu-

ents present and the amount of each can be calculated under any given set of conditions.

Therefore, it should not have been expected that hydrolysis of the solvent in the wheat germ should be complete. It is only because some unknown reaction takes place which removes one of the products of reaction that it can go forward in this manner. That removal reaction is still not understood and, therefore, could not have been expected.

The method claims are limited specifically to the hydrolyzable solvents, and the product claims are limited to the new product obtainable up to date only in this manner.

There is no question as to the value of the invention. There is no dispute that a palatable wheat germ product has long been sought and that a method for making it which would preserve the vitamin B content has also been searched for, for many years. There is no dispute that besides applicant's product and method no such material or method has been found. There is no dispute as to the commercial value and the commercial success of the product. It is rich in vitamins and minerals and completely stable. Mr. Rich is a representative of one of the largest flour mills in Canada and he testified as to the merit and usefulness of applicant's product, as did applicant himself.[16]

The situation, therefore, falls squarely within the rule of the Supreme Court in *Smith* v. *Snow,* 294 U. S. 1 (79 L. ed. 721), and *Paramount* v. *American Tri-Ergon Co.*, 294 U. S. 464, that any invention which is long sought and not found and which accomplishes a highly useful function, is patentable no matter how simple the advance seems.

> "Where the method or device satisfies an old and recognized want, invention is to be inferred, rather than the exercise of mechanical skill." (*Paramount* v. *American Tri-Ergon Corp.*, 294 U. S. at 474.)

16. Rich, appendix pp. 36-37, Record pp. 50-52.

The present development is patentable on any basis. But it was not a simple development. It took years of research and the reasons for its success are still unknown. It was not an obvious step.

Here, as in *Smith* v. *Snow,* 294 U. S. 1, 17, the "history of the prior art serves to emphasize rather than to discredit the striking advance" made by Levin.

It is respectfully submitted that the patent should be issued in the form sought.

The decision of the Board of Appeals of the Patent Office was based on an erroneous interpretation of the facts. It assumed that there was no difficulty in removing last traces of a solvent from a solid food. This basis has been proved unsound and untrue. Such a difficulty exists, and no one except applicant has ever discovered how to overcome it.

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APPENDIX TO BRIEF FOR APPELLANT

FILED OCT 6 - 1941

Joseph W. Stewart

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PRESS OF BYRON S. ADAMS, WASHINGTON, D. C.

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APPENDIX TO BRIEF FOR APPELLANT.

I.

PLEADINGS, DOCKET ENTRIES AND OTHER PAPERS DESIGNATED BY APPELLANT.

1 *Complaint.*

To Enforce Issue of Patent.

Under Section 4915, Revised Statutes of the United States, plaintiff states as follows:

I. The plaintiff, Ezra Levin, is a citizen of the United States and resident of Chicago, County of Cook, and State of Illinois, and brings this suit in his own right.

II. The defendant, Conway P. Coe, is the Commissioner of Patents of the United States, a legal resident of the District of Columbia, and is sued as Commissioner of Patents of the United States.

III. This complaint is filed in accordance with the provisions of Section 4915 of the Revised Statutes of The United States.

IV. The plaintiff, Ezra Levin, filed an application for a patent in the United States Patent Office on August 6, 1936, entitled "Method of Treating Cereal Grain Germ"
2 which was given Serial No. 94,709. A copy of said application and of the file of the patent office relating thereto are hereby proffered. Said application was a continuation-in-part of an earlier application filed by plaintiff on July 31, 1933, which was given Serial No. 683,085, and profert of which is hereby made.

V. Each of said applications, 94,709 and 683,085, was filed in accordance with the laws of the United States and the rules of the Patent Office.

VI. Said application No. 683,085 was duly prosecuted in accordance with the laws of the United States and the rules of the Patent Office until after the filing of said application No. 94,709 on August 6, 1936. Thereafter said application 683,085 was superseded by application 94,709. Said application 94,709 has been duly prosecuted in accordance with the laws of the United States and the rules of the Patent Office.

VII. Said application 94,709 has been passed upon by the Primary Examiner who refused to allow any claim therein, and specifically refused to allow the following claims which he twice and finally rejected:

3. A finished stable, marketable, non-toxic and palatable dry food product with a high content of vitamin B, consisting of all the constituents of natural grain germ except the oily constituents thereof and with such constituents unimpaired and in the same condition as in the natural germ.

4. The process of making a finished stable, marketable, non-toxic and palatable dry food product, consisting in extracting from natural grain germ all of the oil with a heated solvent therefor at a temperature and acidity to maintain the vitamin B content unimpaired, and subsequently completely removing the solvent from the germ.

3 6. A finished stable, marketable, palatable, dry food product consisting of natural grain germ substantially freed from oil by solvent extraction and free of solvent, and having substantially the vitamin B potency of the original germ.

7. A finished stable, marketable, palatable, dry food product consisting of natural grain germ substantially freed from oil by solvent extraction and free of solvent, and having substantially the vitamin B potency of the original germ and having its protease substantially destroyed.

8. The method as set forth in claim 4 in which the solvent is removed from the germ under sub-atmospheric pressure and at a temperature of the order of 125° to 160° F.

9. The method as set forth in claim 4 in which the solvent is ethylene dichloride.

10. The process of making a finished stable, dry, marketable non-toxic and palatable food product consisting of extracting from natural grain germ all of the oil with a heated

solvent of the class consisting of chlorine derivatives of the lower molecular weight paraffins and subsequently completely removing the solvent from the germ at a temperature and acidity to maintain the vitamin B and vitamin B_4 content unimpaired.

11. The method as set forth in claim 10 in which the removal of solvent is accomplished with the aid of steam, whereby the solvent is partially hydrolyzed.

12. A finished stable, marketable, palatable, dry food product consisting of wheat germ substantially freed from oil and having substantially the vitamin B and vitamin B_4 potency of the original germ.

13. A flour comprising a finished stable, marketable, palatable dry food product comprising wheat germ substantially freed from oil by solvent extraction and free of solvent, and having its protease substantially destroyed.

14. The method of treating grain germ to extract therefrom substantially all of the oil which comprises extracting from grain germ substantially all of the oil with a heated solvent hydrolyzing solvent to form an acid under the conditions of the extraction, removing the majority of the solvent by drainage, and removing the last traces of solvent in the presence of heated moisture, wherein the solvent is hydrolyzed to an acid, whereby the entire extraction process is carried out in an acid environment.

4 15. The method of treating grain germ to extract therefrom substantially all of the oil without affecting its vitamin content, which comprises extracting from grain germ substantially all of the oil with a heated solvent which has hydrolyzed to form an acid, removing the majority of the solvent by drainage, removing the last traces of solvent in the presence of heated moisture and under a vacuum at a temperature substantially below the boiling point of water, the grain being maintained in an acid condition during the entire extraction process.

16. A food product comprising a finished, stable, marketable, non-toxic, dry, palatable grain germ material, substantially free from the oily constituents of the grain germ and having the remaining constituents thereof in unimpaired form and having substantially the vitamin B potency of the original germ.

17. The method of treating grain germ to extract therefrom substantially all of the oil without affecting the vitamin content which comprises extracting from grain germ substantially all of the oil with a heated solvent under acid conditions and at a temperature above 125° F. and below 160° F., removing the majority of the solvent by drainage, removing the last traces of solvent in the presence of heated moisture at a temperature between 125° F. and 160° F. whereby the protease in the grain is destroyed, the acid conditions being maintained during the entire extraction process.

VIII. The applicant duly appealed from the decision of the Primary Examiner to the Board of Appeals which affirmed the rejection of the Primary Examiner in their decision of September 25, 1939, as to the claims here in issue. No appeal to the United States Court of Customs and Patent Appeals has been filed.

IX. Plaintiff further states that the invention defined by said claims, and each of them, is new and useful and was not known or used by others in this country before his invention thereof, and was not patented or described in any printed publication in this or any foreign country before his invention thereof, or more than two years prior to his application for patent therefor, and not in public use or on sale in this country for more than two years prior to his application for patent therefor, and not patented in any foreign country by him or his legal representatives on the application filed more than twelve months prior to his application for United States Letters Patent therefor, and not abandoned.

Wherefore plaintiff prays that the Honorable Court decree that the Commissioner of Patents be directed to allow the said claims and to issue a patent thereon.

EZRA LEVIN,
Plaintiff.

By CHRITTON, WILES, DAVIES, HIRSCHL & DAWSON,
Solicitors for Plaintiff,
2800 Board of Trade Building,
Chicago, Illinois.

NELSON J. JEWETT,
850 Munsey Building,
Washington, D. C.,
Phone DI. 2118,
Of Counsel.

* * * * * * * * * *

6 *Answer to the Complaint.*

To the Honorable the Justices of the District Court of the United States for the District of Columbia.

I. Defendant admits the allegations of residence and citizenship of the plaintiff.

II. He admits the allegation of his official position. He admits that his official residence is in the District of Columbia but states that he is a legal resident of the State of Maryland.

III. He admits the allegation of jurisdiction under Section 4915 R. S. (U. S. C., title 35, sec. 63).

IV. He admits that on August 6, 1936, the plaintiff filed in the United States Patent Office an application entitled "Method of Treating Cereal Grain Germ" and that said application was given Serial No. 94,709. He admits that said application is a continuation-in-part of application Serial No. 683,085, filed on July 31, 1933. He admits that plaintiff should furnish duly authenticated
7 copies of said applications at the trial.

V. He admits that each of the said applications Serial Nos. 94,709 and 683,085 were filed in accordance with the laws of the United States and the rules of the Patent Office.

VI. He admits that application Serial No. 683,085 was duly prosecuted in accordance with the laws of the United States and the rules of the Patent Office until the filing of application Serial No. 94,709 on August 6, 1936, and that thereafter said application Serial No. 683,085 was superseded by application Serial No. 94,709 and that the said application Serial No. 94,709 has been duly prosecuted in accordance with the laws of the United States and the rules of the Patent Office.

VII. He admits that said application was passed upon by the primary examiner who refused to allow claims 3, 4, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16 and 17. He denies that the primary examiner has refused to allow any claim in said application Serial No. 94709, and states that claim 18 has been allowed by the primary examiner.

VIII. He admits that plaintiff appealed to the Board of Appeals from the decision of the primary examiner and that the Board of Appeals in its decision of September 25, 1939, affirmed the rejection of claims 3, 4, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15 and 16 but denies that the Board of Appeals affirmed the rejection of claim 17 but states that said claim was held by the Board of Appeals to be allowable to plaintiff. He admits that no appeal has been taken to the United States Court of Customs and Patent Appeals.

8 IX. He admits that plaintiff in his said applications made averments corresponding to the allegations of paragraph IX but denies that these allegations are sufficient to justify the issuance to plaintiff of a patent containing any of the claims 3, 4, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15 and 16, as set out in paragraph VII of the complaint. He denies that plaintiff is entitled to a patent containing any of the claims 3, 4, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15 and 16, as set out in paragraph VII of the complaint since these

claims are deemed to be unpatentable over the following prior patents and publications and for the reasons given in the statement of the examiner in answer to the appeal and the decision of the Board of Appeals, copies of which will be furnished at the trial:

Hoffman,	1,640,193,	August 23, 1927,
Sure,	1,869,721,	August 2, 1932,
Langfeldt,	1,925,489,	Sept. 5, 1933,

Sherman, Chemistry of Foods, 1920, pages 349-350,
Sherman and Smith, "The Vitamines", 1922, page 45.

Profert of copies of these patents and publications is hereby made.

W. W. COCHRAN,
Attorney for Defendant.

February 9, 1940.

I hereby certify that a copy of this Answer was mailed to the attorney for plaintiff on February 9, 1940.

W. W. COCHRAN,
Attorney for Defendant.

* * * * * * * * * *

65 *Findings of Fact.*

1. This is an action under Section 4915 R. S. (U. S. C., title 35, sec. 63) in which plaintiff seeks to have the Court authorize the Commissioner of Patents issue to plaintiff a patent containing claims 3, 4 and 6 to 16, inclusive, of plaintiff's application No. 94,709, entitled "Method of Treating Cereal Grain Germ" which was filed in the Patent Office on August 6, 1936.

2. The claims at bar relate to a food product having a high content of Vitamin B. The product is obtained from the germ of natural grain. Claims 3, 6, 7, 12, 13 and 16 are for the food product and claims 4, 8, 9, 10, 11, 14 and 15 are for the process or method of making the product.

The following claims, 3 and 15, are examples of the claims at bar:

3. A finished stable, marketable, non-toxic and palatable dry food product with a high content of vitamin B, consisting of all the constituents of natural grain germ except the oily constituents thereof and with such constituents unimpaired and in the same condition as in the natural germ.

66 15. The method of treating grain germ to extract therefrom substantially all of the oil without affecting its vitamin content, which comprises extracting from grain germ substantially all of the oil with a heated solvent which has hydrolyzed to form an acid, removing the majority of the solvent by drainage, removing the last traces of solvent in the presence of heated moisture and under a vacuum at a temperature substantially below the boiling point of water, the grain being maintained in an acid condition during the entire extraction process.

3. The patent to Hoffman, No. 1,640,193, August 23, 1927, discloses a method of manufacturing a food product of high vitamin B content. In the method disclosed cereal germs are first freed of their oily content by a suitable solvent such a benzol or carbon tetrachloride. This intermediate product or oil free residue is then subjected to further treatment to produce the final product.

4. The patent to Sure, No. 1,869,721, August 2, 1932, relates to a process of extracting vitamins, including vitamin B, from cereals, and in particular from rice polishings. In the process the vitamin B is extracted from the rice polishings by means of a solvent such as 25% ethyl alcohol and 75% water, to which is added a small amount of an acid. After the extraction of the vitamin B is complete, the solution is distilled off and the vitamin B extract remains. The distillation is carried out under a vacuum of 50 mm to 75 mm pressure and at a temperature of 122° F. to 140° F. The patentee states that the acid preserves and stabilizes the vitamin B and that the distillation at the low temperatures and pressures stated does not destroy the vitamin.

5. The publication "Chemistry of Foods," by Sherman, 1920, pages 349 and 350, discloses that wheat embyro

67 contains some toxic substance apparently associated with the oily constituent thereof, and that extraction of the oil removes in great measure the toxicity of the embyro.

6. The publication "The Vitamins," by Sherman and Smith, 1922, page 45, discloses that vitamin B is destroyed rapidly by alkalies but has relatively marked stability toward hydrochloric acid.

7. The evidence shows that it is common practice to subject solid materials to the action of steam and vacuum to remove the last traces of a solvent from the material.

8. It was not invention to remove the solvent from the intermediate product of Hoffman, by method or process set forth in plaintiff's claims, and omit the last stages of the process set forth in the Hoffman patent.

9. The acid condition favorable to the preservation of vitamin B will obtain in the Hoffman intermediate material when it is subjected to the action of steam to remove the solvent carbon tetrachloride, by reason of the partial hydrolysis of the carbon tetrachloride.

10. Nothing recited in claims 3, 4, 6, 8, 9, 10, 11, 12, 14, 15 and 16 amounts to invention over the prior art.

11. Claims 7 and 13, which are for the product, contain a recitation of the process step "freed from oil by solvent extraction."

68 *Conclusions of Law.*

1. Claims 3, 4, 6, 8, 9, 10, 11, 12, 14, 15 and 16 are not patentable over the prior art.

2. Claims 7 and 17 do not properly define plaintiff's product and are not patentable.

3. Plaintiff is not entitled to the issuance of a patent containing any of the claims at bar.

4. The complaint should be dismissed with costs against the plaintiff.

JENNINGS BAILEY,
Justice.

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69 *Judgment.*

This cause having come on to be heard and having been tried in open court and argued by counsel for the respective parties upon the pleadings and proofs adduced and submitted to the Court.

It is Adjudged this 17th day of June, 1941, that the complaint in this case be, and the same hereby is dismissed with costs against the plaintiff.

JENNINGS BAILEY,
Justice.

Approved as to Form:

NELSON J. JEWETT,
Attorney for Plaintiff.

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71 *Plaintiff-Appellant's Statement of Points Under Rule 73(d).*

Plaintiff assigns the following errors in the record and proceedings in the above-entitled cause:

The District Court of the United States for the District of Columbia was in error as follows:

1. In refusing to authorize and direct the Commissioner of Patents to issue to plaintiff a patent upon the Ezra Levin application Serial No. 94,709, filed August 6, 1936, entitled "Method of Treating Cereal Grain Germ." This application was a continuation-in-part of an earlier application filed by plaintiff on July 31, 1933, Serial No. 683,085.

2. In sustaining and adopting the decision of the Patent Office, holding that the Ezra Levin application did not contain patentable invention.

3. In holding that the Hoffman patent No. 1,431,525, showing a process of malting a wheat germ product from which oil has been removed by solvent extraction from the wheat germ, constitutes sufficient disclosure to justify the rejection of the Ezra Levin application.

72 4. In holding that there was no invention in taking Hoffman's intermediate product and removing the solvent so as to fit it for human consumption.

5. In holding that a process whereby a palatable, stable wheat germ product of high vitamin content is obtained from the residue of organic materials by means of extracting the last traces of solvent from this residue by the chemical action of hydrolysis, which reaction it is disclosed unexpectedly continues to completion instead of being reversible as normally expected, is not invention.

6. In holding that a simple, inexpensive and efficient method of making available for public consumption an estimated $80,000,000 worth of vitamin B complex (present wholesale valuation), did not involve invention.

7. In failing to hold that the solution of a problem such as the one presented here constitutes invention, when to the knowledge of those working in the field the problem has continually existed yet evaded solution by many workers over a period of years.

8. In entering the judgment of June 17, 1941, dismissing the complaint with costs against plaintiff and holding there was no invention.

9. In refusing to enter each and every of the plaintiff's "Findings of Facts" as submitted as follows:

1. The principal basis of the Board of Appeals decision was on a factual matter, as to which additional evidence has been presented here.

2. Wheat is one of the oldest forms of human vegetable food. The wheat kernel consists of three main divisions, the germ, the endosperm, or starch portion, and the bran or hull.

73 3. In former times the whole wheat kernel was used for food and when the wheat was ground into flour, the flour also contained all the elements.

4. The wheat germ, however, quickly became rancid after grinding, and when flour was ground in large quantities

the millers found it necessary to remove the germ, and did so some seventy-five years ago.

5. Some 20-25 years ago it was discovered that vitamin B, existed in wheat germ, and in fact existed there in larger proportions than in any then natural food. Wheat germ contains extraordinarily large proportions of available phosphorus, manganese, iron, proteins, copper and other vitamins of the B complex.

6. Efforts commenced at least twenty years ago to develop wheat germ as a source of these materials and particularly vitamin B. The efforts have been failures except as to applicant's.

7. Some hundreds of millions of pounds of wheat germ are removed each year from wheat in the United States, and are fed to cattle or thrown away. This material contains vitamins and minerals for which the public pays millions of dollars a year in other forms, frequently synthetic. The proteins, minerals and vitamins in wheat germ, if purchased at the drug store in their presently available forms would cost hundreds of millions of dollars per year.

8. The wheat germ is caused to become rancid because of the presence of wheat germ oil. This oil may readily be removed by solvent extraction, which consists in adding an organic solvent such as benzol or hexane to ground wheat germ and then draining off most the solvent. By repeating this action most of the oil may be removed. However, the removal of the last portions of the solvent from the residue is a matter which presented difficulties and was not solved prior to Levin's invention.

9. The same is true of other organic material from which oil is removed by solvent extraction, and which are heat sensitive. Millions of tons of oil are extracted annually in this way from other products, and prior to Levin's invention the fibrous residue was unfit for human food.

74 10. The removal of a volatile liquid solvent from a liquid is a relatively simple matter. In most, but not

all cases, one liquid may be separated from another by distillation, although it is always at the expense of some of each liquid. That is one liquid cannot be removed from another without also removing some of the other. These matters are covered by well known physical laws.

11. The separation of a liquid from a solid, and particularly from a fibrous solid is not governed by known physical laws. The matter is complicated by adsorption and absorption phenomena. For example water, which boiled at 212° F. cannot be entirely removed from an ordinary glass vessel at temperatures far above 212° F. In fact water traces can remain on such a glass vessel at red heat.

12. It takes only a very minute trace of solvent to spoil a food for human consumption. The old example of the spoiling of bread by kerosene vapors, or the spoiling of food in the ice box by immeasurably small traces of vapors from cabbages or other odoriferous materials show this well.

13. Levin solved the problem by using a solvent which had a chemical action by which the last traces of solvent were removed. There is no cited art showing the use of such a principle for the removal of last traces of solvent.

14. The principle employed is that known as hydrolysis. This means a reaction with water which splits up a small proportion of the solvent. The reaction is reversible, that is, a balance or equilibrium is established under normal circumstances so that no matter how small the amount of solvent present, only a small percentage of it will hydrolyze.

15. Levin discovered, however, that in wheat germ, this hydrolysis, instead of being reversible went to completion.

16. Levin used chlorinated paraffinic materials as solvents, and steam as an aid to distillation and these chlorinated materials form chlorides which become stabilized in the wheat germ. The solvents are removed to the last trace.

17. The Hoffman patent shows a process in which oil is removed by solvent extraction from wheat germ. No attempt is made to remove the solvent from the wheat germ.

Instead the germ is given an extensive high temperature treatment, is malted, a water extract separated, and the fibrous residue discarded.

75 18. Hoffman may use a chlorinated solvent, but he did not teach the use of steam or any distillation step in connection with it. His preferred solvent was benzol, where no hydrolysis would have occured.

EZRA LEVIN,

By CHRITTON, WILES, DAVIES & HIRSCHL,
and
N. J. JEWETT,

Counsel for Plaintiff-Appellant.

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II.

EXCERPTS FROM TESTIMONY AND PROCEEDINGS DESIGNATED BY APPELLANT.

21 *Evidence on Behalf of the Plaintiff*

Thereupon **Ezra Levin**, the plaintiff, was called as a witness in his own behalf and, having been first duly sworn, was examined and testified as follows:

Direct Examination

By Mr. Merriam:

Q. Will you state your name, please, Mr. Levin?
22 A. Ezra Levin.

Q. And your address? A. 155 North Clark Street, Chicago.

Q. Are you the applicant in the present case? Are you the plaintiff? A. I am.

Q. Will you state what your academic training has been, Mr. Levin? A. I graduated from Michigan State College in 1914, took my master's degree at the University of Michigan in—

Q. In what? A. Science. —in 1917; wrote a thesis, in part fulfillment of a doctor's degree, which was published. The next phase of my work was teaching high school at Kalamazoo High School and then teaching at the Michigan State College for three years, and then Chief of the Bureau of Agricultural Development in the Michigan State Department of Agriculture for two years.

Q. And what were those years? A. 1921, '22.

Q. And since that time? A. I have been doing research work up until the time that this business developed.

Q. Will you state how you happened to become interested in the wheat germ problem? A. Well, when I was Chief of the Bureau of Agricultural Development in the Michigan State Department of Agriculture the question came up as to the use of various waste products of millers and various waste agricultural products, one of the problems we had in

the Bureau; and one of the waste products was wheat
23 germ, being a by-product of flour mills, something they couldn't do anything with, and my attention was called to that particular problem because I knew at that time that there had already been published that wheat germ was an excellent soure of vitamin B and other valuable materials.

Q. Did you commence work at that time on the problem? A. I did commence work at that time on the problem.

* * * * * * * * * *

24 Q. These exhibits, numbers 3 to 11, Mr. Levin, were prepared by you, were they? A. Yes.

Q. And they show in general the stages of wheat from the beginning of the kernel to the extraction of the oil from the germ and the removal of the solvent from the germ? A. Yes, they do.

Q. Now, will you take them and describe to the Court what each one shows, in order? A. This one is called 3, I imagine. Are these figures your figures and numbers on here? This represents the wheat itself, a picture or section of which is shown here on the board.

(The witness stepped to the blackboard.)

* * * * * * * * * *

25 No. 4. This represents the raw germ. These little flakes that are seen here are represented here in this rather large section (indicating chart), but that is rather out of proportion because the amount of germ that is found in wheat represents only 2 percent of the total wheat, and this may give the wrong impression, in being diagrammatical. It is larger. Naturally, this is much more than 2 percent on the diagram. It is recognized by scientific men and by students that 2 percent is the total amount of wheat germ that is present in the wheat.

To explain that, if I may, wheat germ: "wheat germ" merely means the living, the germinating part of the wheat. The word "germinating" has been cut to the word "germ,"

and it really means the living part of the wheat. When wheat grows, it sends out roots from here and a stem from here (indicating), so that we always know that wheat always grows from the end. That is the living part of the wheat, the wheat embryo.

By Mr. Merriam:

Q. Mr. Levin, how much wheat germ is there in the amount of the flour that is consumed in the United States each year? A. Assuming—

26 Q. Not in the flour but in the wheat from which the flour is made. A. I am assuming that we take the figure of 176 pounds per capita of flour being consumed in the United States. We would assume from that and from the figure I have just given, based on the estimates, that we have about 400,000,000 pounds of this material which is available, which of course, as has been pointed out, is only used in a very small part.

Number 4. I have just shown you that.

Number 5.

Q. Before you leave the wheat germ, Mr. Levin, will you explain to the Court what the chemical content of wheat germ is besides vitamin B1? A. Wheat has truly been called the staff of life, and it is today, and scientific men know, physiologists—we know that that is true, more so today than ever before, in this respect; that wheat contains—that is, the wheat germ contains—more of essentials, nutritional substances, than probably any other foodstuff.

I mean by that that wheat germ is one of the richest sources we have of iron, of phosphorus, of manganese, of copper. I say that because there are probably one or two foods only in the whole category of foods that contain as much as wheat germ does of those substances. We know that wheat germ is one of the richest sources of the vitamin B complex, and the vitamin B complex that is obtained in liquid form is very often made from the wheat germ; we know that—from the natural source, as this is.

Now, the wheat germ also contains protein, and
27 McCullom is the evidence, is the authority, and we have other authorities who state with McCullom that the protein of wheat germ is the finest protein from plant sources. That is, it is equal to that of milk or egg. Now, the wheat germ contains 40 percent of that protein, so that it makes it probably the richest food that we have in protein.

So, all in all, to answer the question, it is one of the richest foods we have in proteins and minerals and vitamin B complex.

Q. Have you made an estimate of how much it would cost to get the same amount of these various minerals and vitamins that you have mentioned in wheat germ if you had to buy them at the drug store? A. Most of the things that we have mentioned are so likely to be lacking in the American diet that we have concentrates of these things now, not only of the vitamin B complex but also of the minerals and also of these special proteins. All in all, we would say this: that of the vitamin B1 alone, $12,000,000 worth of vitamin B1 would be very conservative from a wholesale standpoint; and we would say that on the basis of the synthetic vitamin B1 that is now available $12,000,000 worth of vitamin B1 is found in wheat germ which is cast away at the present time because it can't be maintained stable.

By the Court:

Q. What do you mean by vitamin B complex? What is the meaning of the word "complex"? A. The vitamin B complex is a series of individual vitamins which have heretofore been called vitamin B as a whole. When the nutri-
tionists found that the vitamin B so-called was made
28 up of a number of different things, they gave that general group the name "B complex". They are specifically these:

Vitamin B1 is thiamin, thiamin chloride, which is synthesized and called thiamin chloride.

Vitamin B2 is riboflavin. Riboflavin is synthesized and made and can be bought as such.

Nicotinic acid is the pellagra-preventive vitamin, which of course is very well known now and is being used to prevent pellagra and is found as a salt and is used as such.

We also have pyridoxine, which is the vitamin B6, which has recently been found to be so valuable in a number of things. I won't go into that except to point out, it has been accepted as being valuable nutritionally.

Panthothenic acid, which is an exceedingly important nutritional element.

Then, we have about thirteen other factors which have been found in wheat germs which have not yet been synthesized or are actually isolated, so that it becomes apparent that when we are talking about wheat germs and the vitamin B complex we are talking about a thing of much wider value and importance; and of course in that connection it is evident that the natural product cannot be compared with the synthetic product.

Q. Would you go back to the question I asked about the total value of these materials? A. Well, the total value of all these materials I would estimate would be around in—the amount of germ we throw away would be around $80,000,000, would be a conservative estimate in actual values of these materials.

Q. Is that the drug store value? A. No; that is
29 the actual manufacturing value of substances that are now being made.

Q. What would the retail value be? A. Well, it would be hard to tell, but of course it would be five or six times more than that is, undoubtedly.

Q. Well, you may go on and explain these. This is 5. A. This is the product, as indicated a few moments ago, when it was mentioned that wheat germ had a solvent in it, and the solvent was drained off. This is the product when it is just drained off. Now, when the product is drained off and dried out, as one of the gentlemen mentioned—

Q. This is it (indicating). A. Just dried out. This is it as it is dried out, and it never dries out any more than that is. It is never palatable. That is the way it is dried out in the open. We can leave this in the open a long period of time and it won't dry any more than that.

Mr. Merriam. Does your Honor care to? This is the one with just traces in, and you may smell it. It isn't very much fun.

The Court. I have here (indicating) what contains most of what the witness has been testifying to, and I would say this is not free from odor by any means. I find that if I hold my nose I can take it much better than if I do not.

Mr. Merriam. That is one of the troubles with these synthetic and extracted vitamins, is that you can't get them in consumable form.

The Witness. Simply, may I comment on that?

Mr. Merriam. Yes.

The Witness. The point, the importance of that, of your mentioning that, as far as its economic relationship to the great masses of people that need it is concerned, is this: that that material must be made from this if this material cannot be made solvent-free and palatable, but this material can be utilized as such if it can be made stable.

Mr. Merriam. Incidentally, the package which you have there I believe is a Squibb product, isn't it, your Honor? Yes, it has the Squibb name on it.

The Court. Squibb.

By Mr. Merriam:

Q. And is Squibb the company which owns the Hoffman patent, Mr. Levin? A. The Squibb—well, that is like a rehearsal. Without a rehearsal, I mean. The Squibb Company owns the Hoffman patent and has never used it since they have tried it, because they found it was—well, for some reason or other, which if you would like me to go into it I can do that.

Q. All right. A. They simply have the Hoffman patent, and they don't use it.

Q. Now will you go ahead on these? You have gone up to 7, I think. 8 is the next one. A. This is the oil which is taken out of the wheat germ, and I don't know that you would like to have me say—it isn't pertinent to the discussion at all.

Q. That is a commercial product? A. Simply a commercial product which we take out from this. The only point of interest is that it pays to sell the other material very much cheaper because we have a valuable by-product.

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Q. What is 9? A. 7. You haven't had 7 yet.

Q. Yes, I have. I had 7 and 8. What is 9? A. 9. This is a product which is the raw germ which has not been defatted but just allowed to be, which is sold in the market, and it becomes rancid; and it is a fact that it is the rancidity in wheat germ which makes it noncommercial, and the importance of the whole matter is to get it nonrancid, and so it is ordinarily apparent; it just simply smells like rancid nuts. It might be of interest to you to smell that on that point, like any rancid nut. As a matter of fact, your Honor, the wheat embryo is the nut of the wheat. That is exactly what it is.

Q. That was exhibit 9. Will you explain 10? A. This material is a material that looks like the material we had before but is our material and is six months old. This product is six months old. It has no—we have found at no time—we have had it some places as long as three years old. It doesn't spoil. It does not change in any way.

Q. Now will you identify exhibit 11? A. This is a product which has recently appeared on the market, made by one of the largest solvent-extraction organizations in the world. They make solvent-extracted soy beans, the largest manufacturer in the world to make solvent-extracted soy beans, for cattle food. They have tried to make a material like ours. This is a sample of the material.

Is that all that you would like to have me say?

32 Q. Well, have you tasted that material? Can you tell his Honor whether that is fit to eat? A. The material obviously contains hexane, a solvent which can be determined by testing it. We have bought a number of samples in the open market of solvent-extracted wheat germ, samples of which you have.

Q. Hexane is not a chlorinated solvent, is it? A. No.

Q. Will you explain to the Court how you go about removing the solvent from the wheat germ or removing the oil from the wheat germ and then the solvent from the oil, from the germ? A. The process in general, as we—the process we have gone by, adding—by pouring solvent into raw germ and extracting the oil to less than one-half of one percent. After the solvent is drained off we have two portions, one containing the oil containing the solvent and the residue or the germ containing the solvent. The oil containing the solvent: the solvent is removed by well known methods, and it is understood that it is not difficult to remove the solvent from the oil. The problem of removing the solvent from the residue is a matter of heating it at low temperatures over a long period of time, so that during that long period of time these traces of solvent are changed from the solvent to some other harmless material such as hydrochloric acid. In other words, we hydrolyze the last traces of solvent.

Q. Have you finished your explanation? A. Well,—

Q. Will you explain what hydrolysis is, to the Court? A. This solvent we use contains chlorine as part of its

33 structure, its molecular structure. If we take—

Q. (Interposing) Perhaps it would be better to get a piece of chalk. A. No, I don't think it will be necessary to mark it. If we take this chlorinated solvent, if we take a wheat—that solvent—let me put it this way: That solvent is known by chemists to change in this way: that it is soluble in water, and that portion which is soluble in water may change to hydrochloric acid. That is, to the

extent that it is soluble in water, the chlorine from the ethylene dichloride changes, changes to ethyl alcohol and hydrochloric acid. Now, in wheat germ we have minerals, and these minerals attach themselves to this chlorine to form harmless substances and nonsmelly substances, like iron chloride, for example. Hence we are able to get rid of last traces of solvent. Whereas, in this other material, where they use a solvent that does not have that peculiar chemical phenomenon, they can't get rid of last traces. That is the reason that we use this solvent, and that is the reason we have carried on that type of activity.

Q. Have you made an investigation, Mr. Levin, to determine whether there is any other food material of the fibrous solid nature which has been treated with an organic solvent for solvent extraction and then is fit for human food? A. I have thoroughly seached the literature for ten years, and I have never found a single substance—I know of no substance on the market today that is used for human food that's solvent-extracted, excepting coffee, as I pointed out—as you pointed out, and that is coffee. They use a chlorinated solvent to remove the—that's decaffeinated coffee—to remove the caffeine, and then the coffee is roasted, so of course the trace of the material may or may not be there, but of course its being roasted, it's covered up and we don't know.

34

Q. Coffee isn't being used at this time for food? A. No, it is not used for food, in the sense of a food.

Q. Does the presence of steam in your process enter into the critical nature of the results? A. It does.

Q. In what way? A. Because, being moisture, the steam causes this hydrolysis to take place. That is, it combines with the chlorine to form hydrochloric acid and thus makes it possible to remove the last traces. The proof of the fact that it is the moisture is that we can take hot air—

Mr. Merriam. Just sit down.

The Witness. Beg your pardon.

(The witness resumed the witness chair.)

The Witness. You can take hot air and not get the results that you get with steam. You can heat it, you can dry it, you can do any of the things that you would expect to get rid of these traces of solvent, but we must use steam in order to achieve the result that we want.

Q. When you say you use hot air, what do you mean? You mean in combination with a solvent, do you? A. Yes. In other words, to remove—the cheaper thing would have been to remove—we have tried to remove the solvent with hot air, and we find that impossible.

Q. Well, you have tried chlorinated solvent added
35 with air, have you? A. With air, yes.

Q. And they don't work? A. No, they do not.

* * * * * * * * * *

Q. Mr. Levin, will you state what the relative proportion of vitamin B is in the product which you make, compared to what it was in the original wheat? Germ. Wheat germ. A. Oh, I beg your pardon. You said "wheat." Our product contains more vitamin B than the original wheat germ because it is unimpaired in its processing, so that by taking out the oil and the moisture we concentrate it.

Q. In other words, the proportion of vitamin B is greater? A. Yes.

Q. Although the actual amount is not as great? A. That's right.

Q. Are you familiar with the product of the Hoffman patent? A. Yes, I am familiar with it.

Q. What is the vitamin B content of that relative to yours? A. The vitamin B content of that material is about one-third—less than one-third of that of our product.

Q. Do you know of your own knowledge whether
36 Hoffman made any efforts to remove the solvent from his? A. I do know. Mr. Hoffman told me himself that he never tried to remove all the solvent.

Q. Are you manufacturing a wheat germ product under your patent at the present time? A. Yes, I am.

Q. Under what name is that being made? A. VioBin, V-i-o-B-i-n.

Q. Is it being made by your personally or by a licensee? A. No. It is being made by me, and my company, if you—I don't quite understand the question. Made by me personally.

Q. You are the owner of the VioBin Corporation? A. Yes, I am.

Q. In what form is it manufactured? A. It is manufactured in the granular form, as indicated, and the pulverized form. We pulverize it. There are two forms.

Q. This particular package, which I shall ask to have marked 13, which was shown to the Court earlier,—

(package of VioBin was marked Plaintiff's Exhibit 13 and received in evidence.)

Q. Is that one of your regular trade packages? A. That is one of our regular trade packages.

Q. Is there anything different about that from some of your trade packages? A. It—

Q. Will you look at it and see? A. Oh, no. Oh, this particular—yes, this particular package is a form that is purchased by the Red Cross to be shipped to unoccupied France. It's got a French label on it.

37 Q. For what purpose is it shipped there? A. It is being shipped there because it was determined by the nutritionists of the Red Cross that this product was the one product that would not spoil going across the ocean and would keep in good condition, and they considered it an essential in the substances they have chosen for prisoners and—

Q. I see. A. For prisoners and for women and children in unoccupied France.

Mr. Merriam. Do you want to see that (addressing the Court)? Let me see this.

The Witness. Yes.

By Mr. Merriam:

Q. How long has your VioBin Corporation been making the product? A. Since 1936 we were incorporated.

Q. And what is the experience of that company in the matter of the keeping of the product? A. We have never had a single package of our product ever returned to us, or have we ever had a single complaint of the product having had any solvent in, or any complaint of any kind concerning it.

Q. And for what particular uses is the product employed? A. The product is sold as such in packages. It is sold to add to flour, to add to malted milk, to add to soups, dehydrated soups, various baby foods. It is sold for farina, added to farina, macaroni.

Q. How do your sales of the wheat germ product
38 compare in 1940 to what they were in 1939? A. They are about twice the amount. We sold close to 400,000 pounds of the VioBin in 1940.

Q. How much in 1939? A. Well, about half of that.

Q. State what is the fact as to the financial position of your company as to the amount of capital available for expansion. A. The business has been successful, but the hazard of the business is involved in the possibility of competition which has not yet arisen, and our hope for the future, of course, lies in the fact that we can protect what we have developed.

Q. You said something to me before about shipping to Peru. A. The Peruvian Government, through their Health Department or Health Board, the Department of Health, purchased a quantity of our material to be utilized in the interior of Peru where the Indians have very serious problems of malnutrition, and Dr. Kuychinski, one of the outstanding nutritionists in the world, sent for our material to be utilized in the interior of Peru.

Q. Is your product approved by the American Medical Association? A. The two claims which we make, namely, that the material is richer in vitamin B than the wheat

germ from which it is derived and also the claim that it never deteriorates, are both—the protocols for those claims were submitted to the American Medical Association and passed upon and approved, and the information on
39 that package of the material is information that is accepted by the American Medical Association.

Mr. Merriam. That is all.

Cross-Examination

By Mr. Cochran:

Q. Mr. Levin, I believe you testified that the amount of vitamin B in the Hoffman product is only about a third of what it is in your product. A. The final product.

Q. Yes. Now, how about the Hoffman intermediate product as compared to your product in vitamin B content? A. I wouldn't know that. I have never seen it, and I wouldn't know anything about that.

Q. Well, have you any reason to suppose that it is not about the same? A. Oh, I would assume that the material with solvent in it would have the same amount of vitamin B1. The solvent has no effect on the B1, using the same material.

Q. Now, isn't it true that the solvent used by you or one of the solvents which you have mentioned in your application is carbon tetrachloride? A. That is right.

Q. And that you found that to be very successful? A. Yes, that's successful.

Q. And that is the same solvent which Hoffman uses? A. That is correct.

Q. Now, if the Hoffman intermediate material were subjected to live steam, would that be then your material?

A. Not necessarily, live steam alone. If that inter-
40 mediate product were subjected to the same methods that we use, I would expect that the material would have the same vitamin B content.

Q. Well, isn't it the subjecting of it to live steam—isn't that the treatment which you give it? A. We give it—we

subject it to live steam under—yes, we do subject it to live steam.

Q. To get rid of the solvent? A. Yes, over a certain length of time and under certain temperature. You see, when you ask me one question I don't mean to beg the question.

Q. I see. A. I simply want to add those two things which are involved.

Q. And live steam, if it were forced into the Hoffman intermediate product, would hydrolyze somewhat the carbon tetrachloride? A. I would say that it would, yes.

Q. In that way perhaps bring about an acid condition? A. That's right.

Q. Which would preserve the vitamin B content? A. I would expect that, yes.

Q. Now, is it true that in your process you get rid of all the solvent? A. Yes.

Q. Or, I have in mind asking you about this statement in your application, on page 9 at the bottom. In there you say,

"By the term 'solvent free' as used herein it is
41 meant that the product is palatable and shows no trace of solvent as distinguished by taste or odor."

A. Well, I don't know. I know—I admit that.

Q. Yes. A. I mean I admit the fact that you read it correctly.

(The witness examined the document referred to.)

The Witness. I understand. Yes, I see what you say, but of course I mean by that at least that.

Q. Yes. A. And we do remove the traces of solvent.

Mr. Cochran. I think that is all.

Mr. Merriam. Now, Mr. Levin, just a minute.

Redirect Examination

By Mr. Merriam:

Q. Will you state whether or not it is a fact that taste and odor are the most sensitive tests for solvent that there

are, in food products? A. Yes, they are, but there are also chemical tests.

Q. Are the chemical tests any more sensitive? A. Yes, of course.

Mr. Merriam. That is all.

(Thereupon the witness left the stand.)

Mr. Merriam. May it be understood, your Honor, that all of these exhibits are admitted?

The Court. Very well.

Mr. Cochran. No objection.

Mr. Merriam. Mr. Wurster, will you take the stand?

42 Thereupon **Oscar Herman Wurster** was called as witness for and on behalf of the plaintiff and, having been first duly sworn, was examined and testified as follows:

Direct Examination

By Mr. Merriam:

Q. Will you state your full name, Mr. Wurster? A. Oscar Herman Wurster (spelling).

Q. Your residence? A. 5201 South Kenwood Avenue, Chicago.

Q. What is your occupation, Mr. Wurster? A. I am a chemical engineer.

Q. How long have you been a chemical engineer? A. Well, I have been a chemical engineer for twenty-five, thirty years.

Q. What is your academic background? A. I graduated from the University of Michigan with the Bachelor's degree in 1905 and with the Master of Science degree in 1906 and continued postgraduate studies until February of 1907. Then I went to work in plants.

Q. What company are you now connected with? A. I am the head of the chemical engineering firm of Wurster & Sanger, Incorporated.

Q. And how long has that firm been in existence? A. Since Sanger and I went into partnership in 1921. We incorporated in 1926.

Q. And what is the business of that firm, largely? A. We design and build chemical process equipment,
43 chiefly for the fat and oil and by-product industries, and particularly for the edible oil work, glycerin plants, solvent extraction plants, similar plants for carrying out commercial operations.

Q. Is the preparation and designing of solvent-extraction plants one of the major businesses of that firm? A. Yes, it is one of our branches.

Q. Have you any idea how many solvent-extraction plants you have actually made in the last twenty years? A. Well, I expect six or eight plants of various kinds.

Q. Now will you state, Mr. Wurster, whether in your work in the chemical engineering field you know of any instance in which an organic solid material has been treated with solvent and after the treatment with solvent the material has been then processed to the point where the residue is fit for human consumption? A. No, I don't know of any such case.

Q. If you had been asked in 1933 to design a plant for that purpose, would you have felt that you could have guaranteed success? A. I would say that we would not have undertaken the delivery of such a plant and guaranteed it to produce a residue suitable for human consumption. If such a guarantee had been made, we would have had to insist on first testing it on a pilot plant scale.

Q. Was there any problem in removing solvent from fibrous materials, in general, to a point where the material can be used for animal consumption? A. Yes, that is
44 done right along. It has been done for a great many years. There is a great deal of vegetable oil and also some animal oils are solvent-extracted, and the residues are suitable for cattle feed and poultry and so on.

Q. Can you give us some of those materials, name some? A. Well, taking animal oils first, we extract greases from

meat scraps, and the material extracted is then ground up and made into hog and chicken feed; and in the extraction of cottonseed meal, a great deal of which has been for years sent over to Europe, we press cake. Hydraulic press cake from this country has been sent to Europe and solvent-extracted over there, and the meal has been used for feed; and in this country we have also extracted cottonseed and obtained a residue suitable for cattle feed.

Q. I believe you have told me that in designing and carrying out of solvent extraction process there are several problems involved. Will you just repeat what those problems are? A. Well, there are plenty of very important problems that come up. The ones that we refer to, were in the commercial application of solvent extraction; and that question that you bring up is one that is brought up to us frequently in our commercial work, because chemists work out a solvent extraction process in the laboratory on a laboratory scale, frequently, by what is known as the Soxhlet extraction process, and then think they have something that can be carried out very simply commercially. Well, now, you can do on a small scale in the laboratory, something perhaps which becomes difficult commercially on a large scale. You have got to get out quantities of material, production, so the first important thing is to get that large-scale material in such physical shape that the solvent can permeate it, in the first place; and in the second place, that you can separate the solution from the solid material after you have effected that solution. Now, that is done by flaking the material, or something of that sort, to avoid fine particles. Now, when you have got your solution separated from your solvent material, then you have two other problems:

The simplest one, the first one, is to separate the solvent from the extracted material, fat or oil, which is quite simple.

The next one, then, is to separate the solvent from the solid material, and that is one of the biggest problems in solvent extraction.

Q. Will you state whether or not it is a fact, Mr. Wurster, that so far as solid materials are concerned it is a common practice to subject the materials to the action of steam to remove the last traces of solvent therefrom? A. Yes, that is quite frequent. Steam. Steam and vacuum.

Q. Is that true of food products? A. How? I don't know whether I get your question.

Q. Is that true of food products, that you remove the last traces of solvent, so far that you are able to use them as food products? A. Well, I don't know. I don't know there are any such food products.

Q. Well, I asked you if it was a common practice to do that, and I think you slipped there and misspoke yourself.

A. I don't think I understood your question. There
46 are no common food products that are obtained by solvent extraction.

Q. Is it true as to food products that it is necessary to remove the last traces of solvent in order to make them palatable? A. Yes, it is, because all of the solvents that I know are objectionable to taste.

Q. In removing one liquid from another liquid are there common physical laws which govern the separation? A. Yes. The laws of distillation and fractionation.

Q. In general, if you were asked to separate one liquid from another liquid, is there any considerable problem involved? A. No. That is done. The problem becomes greater as the boiling points of the components approach each other, but it is done regularly, even with liquids whose boiling points closely approach each other.

Q. Is that true of the removal of a liquid from a solid? A. No. In the case of liquids and solids you have got too many other uncontrollable factors entering into it.

Q. Can you name some of those factors? A. Well, you've got questions of surface tension. You've got the solid material. The solvent permeates the solid material, and it won't let loose. The solid material absorbs the liquid material, and it adsorbs it, and you have surface tension there. You have many possible physical differences in the solid.

Q. Can you tell the Court what the fact is as to the difficulty of removing last traces of water in solids? Can you give some examples of that? A. To absolutely remove the final traces of water is very difficult. You've got to
47 heat the material on which you've got a film of moisture very hot, considerably above the atmospheric boiling point of water. You can help to effect that by putting it under vacuum, by using dry agents; but as a matter of fact it illustrates the difficulty. Even on a vitreous surface or a metal surface it is difficult to remove final traces of water. So that it emphasizes the difficulty that there is of taking a liquid out of the pores of a solid material.

Mr. Merriam. That is all.

Cross-examination

By Mr. Cochran:

Q. I believe you testified that it is the common practice to apply live steam to a solid residue to remove the remaining portions of solvent which has been applied to it. A. Yes, steamed off under vacuum. That is the common practice.

Q. Is there any reason—would you have any reason to suppose that the same procedure would not be completely successful if applied to a solid residue of matter which could be used as food? A. I don't think I understand your question, sir.

Q. Would you have any reason to suppose that the application of steam to a residue of food matter would not be successful in removing the solvent from it that had remained after extraction? A. Whether the residue would be suitable for human consumption?

Q. Whether a residue intended for human con-
48 sumption could be successfully treated with live steam to remove solvent. A. It is very difficult to remove it. You even have to use considerable care to make it suitable for animal food, for cattle, so it would be even more difficult to make it suitable for human food.

Q. It is successful, though, is it not? A. I don't know of its having been successful anywhere outside of Mr. Levin's product.

Q. Well, that is what he did; he applied live steam to it; is that it? A. Under special conditions.

Q. Under temperature and pressure, I believe. A. And a certain solvent. It might not apply to all solvents.

(Mr. Cochran. I believe that is all.)

Re-direct examination

By Mr. Merriam:

Q. Mr. Wurster, just to clear this up: In so far as the physical laws of separation go, it does not make any difference whether the product is to be used as a food or anything else, does it, as to how much solvent you can get off? A. No.

Q. The fact is that from any kind of solid residue you don't get the last traces of material off that would be required to be off if you were going to use it for a food; is that correct? A. Yes.

Mr. Merriam. That is all.

49 Recross examination

By Mr. Cochran:

Q. Well, how did they get it off in this case, then, Mr. Wurster? You say they are unsuccessful in getting it out entirely? A. That is correct, by steam. The solvent that Mr. Levin uses is ethylene dichloride, and the final traces remove themselves by hydrolysis, by steaming.

Q. Oh, the steam produces some hydrolysis, and that— A. (Interposing) Yes, and his ethylene dichloride breaks down into hydrochloric acid, which reacts with the mineral products in his wheat germ and increases his chlorides, as shown by analysis.

Redirect examination

By Mr. Merriam:

Q. Just one question: Do you know of any other process of any kind in which hydrolysis is made use of in the same way? A. No, I do not.

(Mr. Merriam. That is all.)

(Thereupon the witness left the stand.)

(Mr. Merriam. Mr. Rich, please.)

Thereupon **Clifford Earl Rich** was called as a witness for and on behalf of the plaintiff and, having been first duly sworn, was examined and testified as folows:

50 Redirect examination

By Mr. Merriam:

Q. Will you state your full name, Mr. Rich? A. Clifford Earl Rich.

Q. And your residence, Mr. Rich? A. In Montreal, Canada.

Q. And what is your occupation? A. I am a chemist.

Q. And are you in the employ of a company in Canada? A. I am the chief chemist for the Ogilvie Flour Mills Company.

Q. How long have you been chief chemist for that company? A. I have been chief chemist since August, 1938.

Q. Are the chemical records of that company in your keeping, under your control? A. Yes.

Q. Will you state what the position of the Ogilvie Company is, in general, in the milling business in Canada? A. The Ogilvie manufacture primarily flour, wheat flour, rolled oats, and breakfast cereals.

Q. Are they one of the largest companies in that field in Canada? A. Yes, one of the largest and the oldest.

Q. Will you state what the experience of that company has been in the manufacture of wheat germ products? A. The Ogilvie Company has known for years that wheat germ is

of excellent food value and has been trying since, I believe, about 1931 to put a wheat germ on the market for human consumption.

51 Q. In the beginning how was that wheat germ product prepared? A. In the beginning they did as most companies tried, to simply package it and sell it.

Q. Without extracting the oil? A. Without, just as it comes from the mill.

Q. What was the result of that program? A. Like all other companies, they had no success because the wheat germ went rancid very quickly, and most of it was returned to the company.

Q. Were efforts made later to control the length of time the product would remain on the shelves? A. Yes. For some time they tried dating the wheat germ. It was dated at the moment that it was packed, and they tried to keep it in no more than a week old, by means of the dates. If it was over a week old it was automatically returned, but their success was very similar.

Q. That is, it was nonexistent? A. They had no success.

Q. Will you state what the company is doing at the present time in respect to wheat germ? A. They have tried a number of experiments to stabilize wheat germ over a period of years and eventually decided to buy VioBin from the VioBin Corporation and to package it and sell it in Canada in that form.

Q. And they are doing that now, are they? A. Yes, they have been doing it for about three years.

Q. Can you give us an idea of the amount that they are purchasing, say, in 1940? A. I remember about three

52 months ago we bought 50,000 pounds, and about a week ago we ordered more from Chicago.

Q. Have you had any complaints as to spoilage on that material? A. No, none whatever.

Q. Do you date the packages on that? A. No, the material comes in hundred-pound bags, and we repackage it just as it comes and keep no records of the date whatever.

Q. I understand that the company made a cereal, a flake product, at one time. Is it a fact that the wheat germ product was included with that? A. Yes. Just prior to my coming with the company or becoming chief chemist a breakfast cereal was put on the market which contained two and a half percent of wheat germ.

Q. Was that defatted? A. No. That was raw wheat germ just as it comes from the flour mill; and the material, the cereal, the new cereal, was practically a failure because rancidity developed if the packages stayed on the store shelves any length of time.

Q. Was that product ever changed in any way? A. When I came with the company or became chief chemist, it was my duty to find out why this cereal was spoiling. I checked all the possibilities there were, and we tried runs containing VioBin instead of the raw germ. Since that time we have had no trouble on that score at all.

Mr. Merriam. That is all.

Mr. Cochran. No questions.

(Thereupon the witness left the stand.)

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Plaintiff's Exhibit No. 2.

173 Endorsed: Filed Jul 18 1941 Charles E. Stewart, Clerk

Department of Commerce

United States Patent Office

To all persons to whom these presents shall come, Greeting:

This is to certify that the annexed is a true copy from the records of this office of the File Wrapper and Contents, in the matter of the Pending Application of Ezra Levin, Filed August 6, 1936, Serial Number 94,709, for Improvement in Method of Treating Cereal Grain Germ.

In Testimony Whereof I have hereunto set my hand and caused the seal of the Patent Office to be affixed, at the City

of Washington, this eighth day of March, in the year of our Lord one thousand nine hundred and forty-one and of the Independence of the United States of America the one hundred an sixty-fifth.

CONWAY P. COE
Commissioner of Patents.

(Seal)

Attest:
D E WILSON
Chief of Division.

* * * * * * * * * *

177 This invention relates to a method of preparing antineuritic products of edible character, such as foods and medicines, and to the products obtained by such method of treatment.

It is known that the so-called vitamins possess various properties and are intimately concerned with the maintenance of bodily processes, and thus are inseparably connected with the normal physiology of the animal, including man. Thus it is commonly accepted that vitamin A functions to prevent certain types of infections, because its absence from the diet results in inflammation of the eyes, the growth of the animal ceases, and if the condition be not corrected, death usually results, the absence of vitamin C from the diet is likely to result in scurvy and hemorrhages; the absence of the vitamin known as water soluble, growth promoting, anti-neuritic vitamin is likely to result in pellagra and neuritis. The existence of an anti-rachitic factor known as vitamin D, whose function is to preserve the normal deposition of calcium in the bones, has
178 been shown. Also the existence of vitamin E which is concerned with the maintenance of the function of reproduction, has been shown.

The present invention is particularly concerned with the so-called anti-neuritic vitamin and the reproduction factor known as vitamin E. It has long been known that yeast contains an abundance of the anti-neuritic factor. However, yeast is not well adapted for use in food or as medi-

cine, because of its highly objectionable odor and taste. It is known that the embryo of cereal grains contain the anti-neuritic vitamin B_1, or B as it is called in the United States. What was formerly taken to be the water soluble, growth-promoting, anti-neuritic vitamin is not an entity but is composed of at least seven different factors, among which are the anti-neuritic factor and the growth promoting and pellagra preventing factor and the anti-paralytic factor B_4 hereinafter described. As yet a uniform terminology for the water soluble vitamin has not been decided upon. British biologists designate the anti-neuritic factor "B_1" and the growth promoting factor "B_2", reserving "B" to identify the complex of the two factors. In the United States it has been suggested that the term "B" be restricted to designate the anti-neuritic factor and that the term "G" be used to denote the growth promoting factor. In the present application the American terminology will be employed and the term "B" will be restricted to refer to the anti-neuritic factor with which the present invention is concerned.

The anti-neuritic factor is present in the embryo of
179 wheat grain, but it is also present in the germ or embryo of other cereal grains such as barley, rye, corn and oats. Heretofore the anti-neuritic factor in cereal grain germs has been but slightly utilized due to the fact that germs tend to turn rancid on standing. The present invention contemplates treating the various cereal grain germs to render them palatable and preservable, thus making the vitamin "B" readily available for use as a food or medicine. To this end the oil in the cereal grain germs is extracted, leaving a wholesome and preservable product.

Referring again to the cereal grains, it has been shown that vitamin E exists abundantly in the germ oil of these grains, particularly wheat, corn and oats. A further object of the present invention is to remove the oil from the grain germs without damaging the vitamin E content.

A further object is to devise a method whereby cereal grain oil may be removed from the germ without destroying

the vitamin B in the new cereal product and to preserve the vitamin E content in the germ oil.

The problem of how to treat cereal grain germs in such manner that the vitamin B content of the germ is not destroyed has been the subject of intensive and careful investigation but no thoroughly satisfactory solution of the problem has heretofore been offered. It has long been thought that the application of heat readily destroys the anti-neuritic factor. I have discovered that by extracting the oil from the grain germ in an acid condition the application of heat during the treatment does not impair
180 the vitamin B content of the product and the germ oil extracted may be employed as a source of vitamin E. Moreover, it is possible in this operation to achieve these results without an appreciable solvent loss.

This invention will be readily understood from the following description and the accompanying drawing, which illustrates more or less diagrammatically an arrangement of extractor, vaporizer, condenser, and receiver, which has been found well suited to the practice of the invention.

Referring to the drawing, 4 indicates an extractor supporting a removable foraminous basket 5 adapted to contain the cereal germ to be treated, and open and closed steam coils designated by the numerals 6 and 7, respectively, located below the basket. An agitating device (not shown) may be employed in the extractor if desired. The head 8 is securely fastened to the extractor by bolts or otherwise, in such manner that it may be readily removed.

A vapor pipe 9 provided with a valve 10 leads from the top of the extractor to the upper end of the water-cooled coil of a condenser 11; and from the bottom of the coil leads through a separator 12, a pipe 13 for conducting the condensed solvent to a receiver 14 supported in an elevated position above the extractor when supplied with a vent 15 for releasing uncondensible gases.

181 At 16 is shown, leading from the top of the separator 12, a valve drain for drawing off the watery product of condensation. From the bottom of the receiver a

pipe 17 equipped with a shut-off valve 18 leads to the bottom of the extractor.

From the top of the extractor a pipe 19 provided with a check valve 20 and control valve 21 leads to a vaporizer 22 supported in upright position and having rounded top and bottom. At 23 and 24 are shown, respectively, open and closed steam coils preferably located in the bottom of the still. A draw-off line for the oil is indicated at 25.

The vapor line 26 leads from the top of the vaporizer to the upper end of the water-cooled coil of a condenser 11′ which is similar in construction to the condenser 11. In like fashion the lower end of the condenser coil leads into the receiver 14 after passing through the separator 13′ equipped with a water drain 16′.

A drain pipe 27, provided with a control valve 28, is tapped into the bottom of the extractor and forms a connection with the pipe 19.

The operation of this apparatus is as follows:

A charge of cereal grain germ, from which it is desired to remove a maximum quantity of oil, is placed in the foraminous basket and introduced in to the extractor and the head bolted down. The solvent is then allowed to flow from the receiver into the extractor until it is just below the level of the pipe 19. Steam is turned on in the closed steam coil and the mixture of solvent and grain germ heated. It is desirable to maintain a temperature in the extractor somewhat below the boiling point of the solvent, preferably below 170° F. The oil readily dissolves in the hot solvent and the light solution rises to the top of the extractor and overflows through the line 19 past the check valve 20 into the still. As the oil in solution passes out at the top of the extractor, additional solvent flows in the bottom. Cereal grain germ contains varying amounts of water, and a solvent having a specific gravity heavier than water is particularly helpful in displacing water from the germ.

In operation, the still is maintaned at temperatures higher than the boiling point of the solvent, and when the solution

of solvent and oil enters the still, the solvent is vaporized. The check valve prevents the vapors from back-flowing into the extractor and they rise through pipe 26, are condensed in 11′, and flow into the receiver, to be put through the system again. When a maximum amount of oil has been extracted and is completely in solution, the liquid contents of the extractor are withdrawn through the drain 28. The valves are then closed and additional steam applied
183 in the closed steam coil, vaporizing the solvent clinging to the grain germ, returning the solvent to the receiver by means of the pipe, the condenser 11, and the water separator. The last ends of the solvent are completely removed from the grain by a blast of live steam which completely vaporizes the occluded solvent and whatever water may remain in the extractor. The water is separated and withdrawn from the system.

The grain germ may be dried in the extractor before removal by the addition of an air line. The solvent collecting in the still is completely vaporized by the application of additional closed steam, finishing up with live steam. After the solvent has been vaporized and more or less completely passed out of the still, the oil is removed from the still through the withdrawal line. I prefer to operate my process in a closed system in order that the vitamin E in the germ oil may not be subjected to oxidation.

After the withdrawal of the grain germ and the germ oil, the solvent is concentrated in the receiver and the apparatus is ready to receive another charge of grain germ. After extraction, the grain germ is light, dry and of a floury consistency and has been found to have a high mineral, protein, and water-soluble vitamin content. Among other things, it is adapted to be used as a supplement to cereal foods, as a liquid extract containing vitamin B, and may be employed in baking as a supplement to flour. The extracted germ oil is also a valuable product in that it is rich in vitamin E.

184 It is preferred that the steaming process in the extractor be operated under a substantial sub-atmos-

pheric pressure. The degree of vacuum will vary during the process according to the solvent used, but it is preferred that the vacuum be such that the temperature in the extractor does not exceed 160°F. and does not fall below 125° F. This temperature range is particularly important where the product is to be used as a flour or in connection with baking. The wheat germ contains a proteolytic enzyme presumably protease, which unless destroyed attacks the gluten of the flour and causes hard and unsatisfactory baked products. This enzyme is not destroyed by the extraction temperature even up to temperatures of 170° F., presumably because of the absence of moisture. However, in the presence of steam the enzyme can be destroyed at the lower temperatures designated.

In operating the vacuum extraction the pressure is held at the desired point to give the desired temperature until the temperature starts to rise upon substantial elimination of the solvent. At that point the pressure is lowered and live steam introduced at approximately the same temperature range. This operation is then continued until the product is solvent free.

By the term "solvent free" as used herein it is meant that the product is palatable and shows no trace of solvent as distinguished by taste or odor.

185 Where it is not desirable to destroy the proteolytic enzyme, temperatures or other conditions may be employed which will preserve the enzyme.

Of course, when a vacuum is employed a vacuum pump is added to the apparatus shown in the drawing.

As is readily seen, my process is a batch operation in a closed system, and has only an imperceptible solvent loss.

While ethylene dichloride is a preferred solvent, any petroleum hydrocarbon solvent boiling below 120° C. and carbon disulfide are suitable for my purpose, if a weak acid is added to the solvent to maintain an acid condition in the extractor. It is important that an acid condition be maintained during the extraction. The foregoing solvents

are inflammable and consequently I particularly desire to employ the various chlorine derivatives of ethane such as dichlorol-ethylene, trichlorol-ethylene, perchloral-ethylene tetrachloral-ethane, and pentachloral-ethane. These solvents tend to hydrolyze when contacted with the moisture-containing cereal grain germ and it is not necessary to add acid when they are employed. It is apparent that the solvent will be hydrolyzed from the beginning of the extraction until the solvent is drawn off, that is, from the time the solvent contacts the moisture in the grain germ until
186 it is driven off with steam. Excellent results have also been obtained with carbon tetrachloride.

The hydrolysis of such a solvent as ethlene di-chloride is of material assistance in removing the last traces of solvent during the steaming operation.

The treated wheat germs are preferably not completely freed from oil but enough of the oil is retained so that the material will not become rancid or unpalatable. Inasmuch as the oil contains some vitamin E and A the retention of it in palatable quantities is desirable.

The above treatment also may be employed to adjust the moisture content of the wheat germ. In all cases the finished product is thoroughly dry and contains less than 10% of moisture, normally about 7%.

In some instances, it has been found desirable to employ the entire grain seed as a charge. In this case, it is desirable to grind the grain seed before it is inserted in the extractor.

187 The treatment hereinbefore described, when employed with the preferred solvents, and particularly with ethylene di-chloride, extracts the oil from the wheat germ without damaging the vitamin B_4 content thereof. This vitamin is found in liver and also in wheat germ and is of great value in the treatment of pernicious anemia. When the oil of the wheat germ is extracted with hexane, ether or acetone, the vitamin B_4 is dissolved out and lost, whereas in the present method of procedure the vitamin

B_4 is retained and the finished dry product is as high as, or higher, in the vitamin than liver.

While I have described in considerable detail the specific form of apparatus which I find well adapted to the carrying out of my process, as well as the details of the particular commercial process in accordance with the invention, it will be understood that this is illustrative only and for the purpose of making clear the nature and mode of applying the invention and that the invention is not to be regarded as limited in scope to the illustrative details given, except insofar as such limitation is included within the terms of the accompanying claims, in which it is my intention to claim all novelty inherent in my invention as broadly as possible in view of the prior art.

This application is a continuation-in-part of my copending application, Serial No. 683,085, filed July 31, 1933.

188 What I regard as new and desire to secure by letters patent is:

1. A finished stable, marketable and palatable dry food product consisting of wheat germ substantially freed from oil.

2. A finished stable, marketable and palatable dry food product consisting of wheat germ substantially freed from oil and having substantially the vitamin B potency of the original germ.

3. A finished stable, marketable, non-toxic and palatable dry food product with a high content of vitamin B, consisting of all the constituents of natural grain germ except the oily constituents thereof and with such constituents unimpaired and in the same condition as in the natural germ.

4. The process of making a finished stable, marketable, non-toxic and palatable dry food product, consisting in extracting from natural grain germ all of the oil with a heated solvent therefor at a temperture and acidity to maintain the vitamin B content unimpaired, and subsequently completely removing the solvent from the germ.

5. A finished stable, marketable, palatable, dry food product consisting of wheat germ substantially freed from oil by solvent extraction and free of solvent.

189 6. A finished stable, marketable, palatable, dry food product consisting of natural grain germ substantially freed from oil by solvent extraction and free of solvent, and having substantially the vitamin B potency of the original germ.

7. A finished stable, marketable, palatable, dry food product consisting of natural grain germ substantially freed from oil by solvent extraction and free of solvent, and having substantially the vitamin B potency of the original germ and having its protease substantially destroyed.

8. The method as set forth in claim 4 in which the solvent is removed from the germ under subatmospheric pressure and at a temperature of the order of 125° to 160° F.

9. The method as set forth in claim 4 in which the solvent is ethylene dichloride.

10. The process of making a finished stable, dry, marketable non-toxic and palatable food product consisting of extracting from natural grain germ all of the oil with a heated solvent of the class consisting of chlorine derivatives of the lower molecular weight paraffins and subsequently completely removing the solvent from the germ at a temperature and acidity to maintain the vitamin B and vitamin B_4 content unimpaired.

190 11. The method as set forth in Claim 10 in which the removal of solvent is accomplished with the aid of steam, whereby the solvent is partially hydrolyzed.

12. A finished stable, marketable, palatable, dry food product consisting of wheat germ substantially freed from oil and having substantially the vitamin B and vitamin B_4 potency of the original germ.

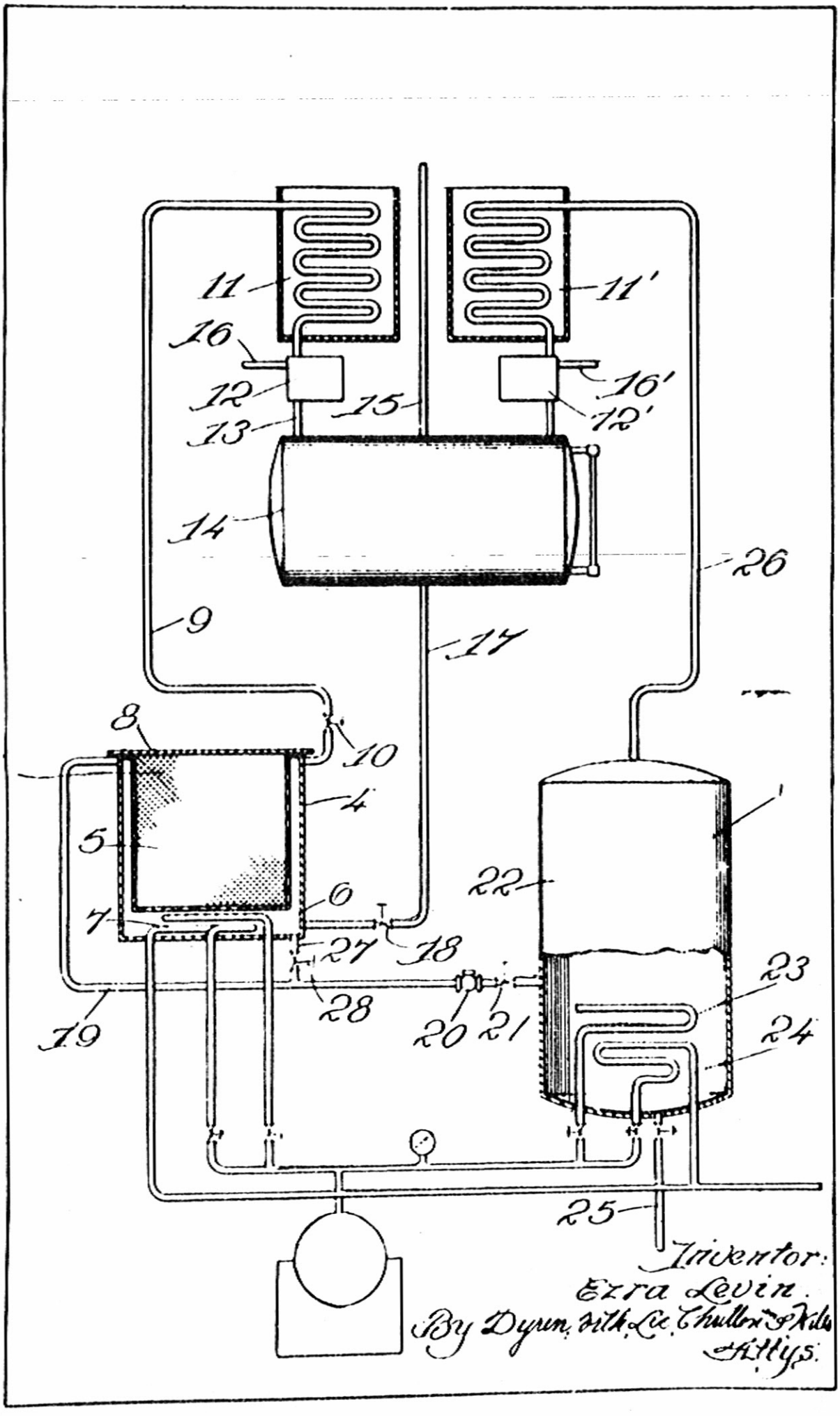
13. A flour comprising a finished stable, marketable, palatable dry food product comprising wheat germ substantially freed from oil by solvent extraction and free of solvent, and having its protease substantially destroyed.

14. The method of treating grain germ to extract therefrom substantially all of the oil which comprises extracting from grain germ substantially all of the oil with a heated solvent, a hydrolyzing solvent to form an acid under the conditions of the extraction, removing the majority of the solvent by drainage, and removing the last traces of solvent in the presence of heated moisture, wherein the solvent is hydrolyzed to an acid, whereby the entire extraction process is carried out in an acid environment.

191 In testimony whereof I have hereunto set my hand and seal this 29th day of July, 1936.

EZRA LEVIN. (Seal)

* * * * * * * * * * * * *



Inventor:
Ezra Levin.
By Dyer, Tibbitt & Le Chulton & Wilkinson
Attys.

194 Art cited:

Hoffman,	1,640,193,	Aug. 23, 1927,	99/27
Sure,	1,869,721,	Aug. 2, 1932,	167/81
Sure,	2,015,876,	Oct. 1, 1935,	X99/11
Matio, (Br.)	362,023,	Dec. 3, 1931,	167/81
Agopian,	1,468,251,	Sept. 18, 1923,	167/V
Dietz,	1,974,808,	Sept. 25, 1934,	99/V

Chemistry of Foods and Nutrition. Sherman, 1920. pp. 349/350. (Copy in Div. 63)

The Vitamins. Sherman & Smith, 1922. Page 45. (Copy in Div. 63)

Biological Chemistry Journal, Vol. 37, 1919. Page 569. (Copy in U. S. Pat. Off. Library)

Rappaport (Br.), 5,270 of 1887, 87/14

Cregor, 1,646,182, Aug. 23, 1927, 99/27

All the claims are rejected as lacking invention over Sure, Dietz, or Hoffman in view of the remaining art cited above. Sure and Sherman & Smith definitely teach the fact that "acid tends to stabilize the vitamin B in wheat embryo." It appears obvious that if Sure did not intend to extract water soluble vitamin B, he would not employ a solvent mixed with water. Note lines 83 to 89 of Dietz, who teaches the use of merely ordinary fat solvents in extracting the oils from wheat germ but leaves the vitamin B in the germ. Hoffman extracts oils from wheat germ by the use of solvents such as carbon tetrachloride, the latter hydrolyzing to form an acid condition under the influence of heat in similar manner as claimed by applicant. The additional steps of further treating the extracted wheat germ by Hoffman are additive in character and do not detract from the pertinency of said reference in regard to the
195 use of an acid hydrolyzable solvent to extract the oils from wheat embryo without effecting its vitamin B content. The product resulting from the process steps outlined above would necessarily anticipate the product defined herein.

Claims 1 to 3 and 12 are additionally rejected as fully met by the reference in the Journal or the text book cited above, wherein there is recited "an oil with definitely toxic properties has been extracted from wheat embryo." Obviously the remaining wheat germ may be used for its residual known edible food or nutritive values, as shown in Sherman, cited above.

Claims 5, 6, 7, and 13 are additionally rejected upon the ground that a product should not depend for patentability upon the process step or steps of making the same, "freed from oil by solvent extraction" being held such a process step in the claims. See Westinghouse Elec. Mfg. Co. v. Quackenbush 53 F (2) 632.

All the claims stand rejected.

A. H. WINKELSTEIN
Examiner.

196 Hon. Commissioner of Patents,
Washington, D. C.

Sir:

In the above application kindly enter the following amendment:

Cancel Claims 1 and 2 without prejudice.

Claim 4, last line, after "subsequently" insert—completely—.

Cancel Claim 5 without prejudice.

Claim 6, line 2, change "wheat" to—natural grain—.

Claim 7, line 2, change "wheat" to—natural grain—.

Claim 10, line 5, after "paraffins" insert—and subsequently completely removing the solvent from the germ—; line 7, change the comma after "unimpaired" to a period and cancel the remainder of the claim.

197 Claim 13, line 2, change "consisting of" to —comprising—.

Claim 14, line 4, cancel the comma.

Add the following new claim:

15. The method of treating grain germ to extract therefrom substantially all of the oil without affecting its vita-

min content, which comprises extracting from grain germs substantially all of the oil with a heated solvent, which has hydrolyzed to form an acid, removing the majority of the solvent by drainage, removing the last traces of solvent in the presence of heated moisture and under a vacuum at a temperature substantially below the boiling point of water, the grain being maintained in an acid condition during the entire extraction process.

Remarks

Reconsideration and allowance of the application as amended are respectfully requested.

As stated at the interview courteously extended by the Assistant Examiner in charge of the application, the present case defines in a number of points in addition to the features pointed out in the parent application.

The argument of the Examiner in the prosecution of the parent case and in this one has been to the effect that there was no invention in preparing a vitamin containing a palatable non-toxic grain germ product over the references which purport to show that the oil in grain germ is toxic and which further indicate that Vitamin B is stable in an acid environment. The Examiner therefore argued that all that was necessary was that wheat germ be treated with an oil solvent in an acid condition and that the experimenter would then have a perfectly suitable edible product.

198

The fact that so much work had been done on wheat germ without any one's even suggesting that it could be used for a vitamin food apparently did not suggest to the Examiner that perhaps there might have been some difficulties which prevented these investigators from using the material.

If there had been no difficulties in the way of using wheat germ, why does the Examiner think that Hoffman malted his intermediate product? If he had thought that the grain germ itself could have been used for food he certainly would not have carried on his process in the expensive way

he does. We understand that his final product actually has about one-third the Vitamin B content of applicant's.

The main difficulty which was encountered was of course the removal of the solvent without destruction of the vitamins. Wheat germ is a complex, somewhat fibrous material containing oils, some moisture, protein and other material. The extraction of solvent from such a material is by no means the simple process which the Examiner appears to think it is. We fail to think of any food product of
199 such a complex nature which has in its preparation been treated with a solvent such as carbon tetrachloride. It is of course easy to remove the bulk of the solvent, but the last traces are extremely difficult to remove without destroying the sensitive vitamins.

We therefore think it clear that the reason Hoffman had to go to his expansive malting process was that he knew of no way to make wheat germ palatable after treating it with benzol or carbon tetrachloride.

The Dietz reference makes no showing whatsoever as to a method for freeing the germ from solvent, nor does he show how to produce a material containing the vitamins. His specific solvent, ether, is not suitable because it removes the vitamin B-4 from the wheat germ.

These are the only two references which show the removal of oil from the wheat germ.

Applicant is not only the first to produce this substance but he is also the first to show how to do it. Applicant's product is a palatable solvent-free material having as high or higher Vitamin B content than the original germ (the product is relatively higher in Vitamin B because of the removal of oil).

Applicant manages to secure this product by using a solvent such as ethylene dichloride which hydrolyzes during the extraction of the oil to produce an acid and thereby maintains the vitamin, and further by extracting below the boiling point of water and under a vacuum which
200 likewise is necessary to the preservation of the vita-

min, and is enabled to extract the last traces of solvent readily because of the hydrolyzing thereof.

The importance of this latter point is indicated by the fact that the final product shows the presence of chlorine even though all of the solvent has been removed.

Such a product is clearly not anticipated by Hoffman for the reasons already pointed out. In the first place Hoffman shows benzol and carbon tetrachloride as equivalents for his process, and therefore he clearly was not interested in maintaining the vitamins. Moreover, he shows no method of extracting the solvent or extracting it completely and inasmuch as he cooks the product under a presure of 15 lbs. per square inch he cannot be assumed to have gone to the trouble of extracting under a vacuum to preserve the vitamins. As we have already pointed out, it seems quite apparent that his malting and cooking operations were used because he could not free the product from solvent to an extent necessary to render it palatable.

Claim 8 is specific to the use of sub-atmospheric pressure and temperatures of the order of 125° to 160°. This process is not indicated in any of the references.

Claim 13 and Claim 7 specifically point out a further improvement of the present process. Wheat germ *per se* is entirely unsuitable for flour because it forms hard lumps in the baked product. Applicant has discovered that this is due to the presence of protease and has further discovered that the protease can be destroyed at low temperatures after the extraction treatment by the use of steam, even though it was not destroyed at higher extraction temperatures in the absence of steam.

201

There is no suggestion of a process for this purpose in any of the references, and these claims clearly distinguish therefrom.

Claim 9 is specific to the use of ethylene dichloride as a solvent and this is not shown in the references.

Claim 15 has been added to point out in greater detail the extraction method actually followed and this likewise differs from the references.

The claims like 13 and 7 define the product as one freed from oil by solvent extraction. This terms is inserted in these claims to provide a basis for the term "free of solvent". Applicant is unable to think of a better way of pointing out this characteristic of the product, but if any better form can be suggested we will be glad to change. In the absence of any other way of claiming the product it is of course perfectly proper to define it by the method of production.

The arguments already made point out the inapplicability of the Examiner's contention that the Journal and textbook references meet the present case. The mere fact that the oil in the germ is toxic does not indicate that the remaining germs is suitable for food, nor does it indi-
202 cate that there is any way of extracting the oil without injuring the vitamin content and still leave the product suitable for food purposes.

Respectfully submitted,

DYRENFORTH, LEE, CHRITTON & WILES
Attorneys.

Chicago, Illinois,
CJM:DP
April 26, 1937.

* * * * * * * * * *

203 Responsive to amendment of Apr. 28, 1937.
Claims are 3, 4, and 6 to 15.

Claims 3, 4, and 6 to 14 are again rejected as lacking invention over the art for the reasons of record. Claim 15 is now rejected on the same art and for the same reasons.

The art clearly discloses the valuable properties of wheat germ. It is known that the germ contains valuable vitamins. It is also known that the oil may be extracted. It is well recognized (applicant's arguments to the contrary notwithstanding) that wheat germ is a vitamin food. The art shows this. Hoffman and Dietz and Sherman also show it.

Hoffman malted his intermediate product to obtain addi-

tional changes. The omission of the Hoffman steps with their known functions does not involve invention.

As to Dietz, the mere selection of a solvent suitable for extracting particular parts of the germ does not involve invention. In this connection, the claims do not avoid the ether of Dietz. Vitamin "B" is a complex, and it is not definite from applicant's disclosure that any particular vitamin B fractions are retained or that other fractions are lost. Moreover, the carbon tetrachloride shown by Hoffman certainly extracts the germ in the same manner as in applicant's process. In this connection, to remove all of the solvent is not only obvious, but would be followed by those carrying
204 out the Hoffman or Dietz processes. ~~At most, the~~ difference would be only one of degree.

The other features of steam use, or use of ethylene dichloride, etc. do not impart patentability.

This action is Final.

A. H. WINKELSTEIN,
Examiner.

* * * * * * * * * *

205 Honorable Commissioner of Patents,
Washington, D. C.

Sir:

In the above application kindly enter the following amendment:

Claim 14, line 4, after "hydrolyzing" insert—solvent to form an acid—; same line cancel—to an acid—.

Claim 15, line 4, after "solvent" insert—which has hydrolyzed to form an acid; line 5, cancel—under acid conditions—.

Remarks

Reconsideration and allowance of the application as amended are respectfully requested.

Withdrawal of the final action of the Examiner is requested in view of the interview recently courteously extended, at which the Examiner stated that he wished to cite additional art against the method claims.

Applicant is also filing an affidavit carrying his date back of the Dietz reference. It will be noted that the present application is a continuation-in-part of an earlier application filed July 31, 1933 which antedated the issue date of the Dietz patent.

206

Articles have just come to applicant's attention in the "Proceedings of the Society for Experimental Biology and Medicine", Vol. 36, No. 3, April, 1937, Pages 424-427. These show work done by five individuals in Philadelphia demonstrating positively the sarcomogenic properties of wheat germ oil. The Examiner's attention is particularly directed to the first paragraph of the first article in which it is stated:

"All animals fed this preparation of wheat germ oil eventually develop tumors."

Reference is also made to the article of E. V. McCollum and others on Dietary Deficiencies of Wheat Embryo, Pages 105-127, Journal of Biological Chemistry, Vol. XXV, No. 1. These researchers determined that (Page 115):

"There is contained in the wheat germ a substance which is distinctly toxic to animals. This is in great measure removed by extraction with ether, and is found in the fat fraction."

These articles clearly sustain applicant's contention that wheat germ has been considered dangerous from a food standpoint.

With respect to the reference Hoffman, there is no showing of applicant's product in the reference. Hoffman never obtained applicant's product and there is no suggestion in his patent that would lead one to follow applicant's steps to obtain it.

207

Hoffman did not consider his material worth utilizing and believed he was forced to malt it to make it fit for anything.

Such an intermediate product in a prior patent is not applicant's, and even if it were, the law is clear that it would not constitute a reference. See Knee v. Karmin, 274 Fed. 720 at 723, where the court said:

'It is quite clear from his own statement that this was never an article of manufacture; it was but a step in a process of his own to make something else, and I think it does not come under the requirement of the statute, that the article must be either in public use or offered for sale.''

A publication relating to an article unfit for use or sale is no better than the use of the article itself.

With respect to the Examiner's contention that the removal of all of the solvent is an obvious step, we admit that the desire to remove all solvent might be obvious in many cases, but in the present case, there was no known method for so doing which did not damage the product. Applicant is the first to teach how to do it, and the steps given in the method claims are those requisite for such purpose.

Respectfully submitted,

DYRENFORTH, LEE, CHRITTON & WILES,
Attorneys

Chicago, Illinois
January 12, 1938

* * * * * * * * * *

208 *Affidavit*

STATE OF ILLINOIS,
County of Cook, ss:

Ezra Levin, being first duly sworn, on oath deposes and says that he is the applicant in the above entitled application and that prior to February 16, 1931, he completed the invention of the above entitled application by preparing a finished, stable, marketable, non-toxic, and palatable dry food product from whole wheat germ, the final product having a high content of vitamin B and containing substantially all of the constituents of the natural grain germ, except the oily constituents.

Applicant prepared such product by freeing the wheat germ from oil by solvent extraction with a chlorinated oil solvent and subsequently freed the wheat germ from the

solvent. The solvent extraction was carried out under acid conditions.

Affiant further states that he does not know and does not believe that the invention has been in public use or on sale in this country or patented or described in a printed publication in this or in any foreign country for more than two years prior to his application Serial No. 683,085, filed July 31, 1933, of which this application is a continuation-
209 in-part, as to anything shown in said application, or for more than two years prior to the filing date of this application as to anything disclosed in this application, which is not disclosed in said parent application; and affiant further states that he has never abandoned the invention.

EZRA LEVIN.

Subscribed and sworn to before me this 10th day of January, A. D. 1938.

IRENE JEDLICK,
Notary Public.

My Commission Expires Jan. 11, 1939

* * * * * * * * * *

210 Responsive to amendment of Jan. 14, 1938.
Added art:
Langfeldt, 1,925,489, Sept. 5, 1933, 167/81

The final rejection has been withdrawn to permit a reconsideration of the claims in view of the newly discovered reference cited above. The amendment and affidavit of January 14, 1936, have been entered.

The Dietz reference is not withdrawn, since the affidavit does not give sufficient data and evidence to comply with Rule 75. In re Garratt, 431 O. G. 777.

As to applicant's original application filed July 31, 1933, that date does not precede the filing date of the Dietz patent. The patent is not withdrawn.

Applicant's remarks have been carefully considered. However, no reason is seen for withdrawing the rejection previously of record. The removal of all of the solvent is an obvious procedure. In this connection see Langfeldt who

appears to remove all of the solvent in a vitamin extraction process.

The product does not distinguish in a patentable sense over the products of the references.

This action may be taken as final for purposes of appeal.

A. H. WINKELSTEIN,
Examiner.

* * * * * * * * * *

211 Hon. Commissioner of Patents,
Washington, D. C.

Sir:

In the above application kindly enter the following amendment:

Add the following new claims:

16. A food product comprising a finished, stable, marketable, non-toxic, dry, palatable grain germ material, substantially free from the oily constituents of the grain germ and having the remaining constituents thereof in unimpaired form and having substantially the Vitamin B potency of the original germ.

17. The method of treating grain germ to extract therefrom substantially all of the oil without affecting the vitamin content which comprises extracting from grain germ substantially all of the oil with a heated solvent under acid conditions and at a temperature above 125° F. and below 160° F., removing the majority of the solvent by drainage, removing the last traces of solvent in the presence of heated moisture at a temperature between 125° F. and 160° F. whereby the protease in the grain is destroyed, the acid conditions being maintained during the entire extraction process.

212 Remarks

Reconsideration and allowance of the application, as amended, are respectfully requested.

A new affidavit under Rule 75 is being filed herewith which disposes of the Dietz patent as a reference.

We are also filing herewith a copy of an article by Schlutz and Knott showing the effect of Vitamin B upon the appetite of children. As will be seen from Page 414, Vitamin B was given in the form of stabilized wheat germ prepared by the VioBin Corporation in accordance with the present application.

We are also filing herewith a small folder of Baur Flour Mills, St. Louis, which incorporates applicant's product with its flour.

We are also filing herewith a folder of VioBin Corporation giving some of the uses to which the product has been put and the authority for the value of the product for these purposes. As will be noted from this folder, the product has been accepted by the American Medical Association.

We are also filing copies of two letters, one dated September 18, 1936, the other May 9, 1938, from C. A. Elvehjem of the College of Agriculture, University of Wisconsin, indicating the high iron and Vitamin B content of applicant's product.

213 We are also filing herewith a copy of a letter from Dr. Steck of the University of Illinois College of Medicine showing the use of the product in connection with arthritis.

From the standpoint of patentability of the present invention, there are several points upon which we conflict with the Examiner's opinion.

The first is with reference to the general value of wheat germ as a vitamin food. The Examiner states that the art, which now consists of Hoffman and Sherman, shows this to be true. We have pointed out in our previous amendments that Hoffman does not show the use of wheat germ as a food. On the contrary, Hoffman was forced to malt his product in order to make it available for any purpose. Sherman merely quotes McCollum who uses wheat embryo as a source of Vitamin B.

If we check back to the original McCollum work, however, we find that McCollum definitely states that wheat embryo

is toxic. Reference was made to the McCollum article in the last amendment. This article appears in Journal of Biological Chemistry, Vol. XXV, No. 1 Page 105 and the particular reference will be found at Page 115. We also have
214 referred to the proceedings of the Society for Experimental Biology and Medicine, as showing the sarcomogenic properties of wheat germ oil. Furthermore, at the interview courteously extended by the Assistant Examiner in charge of the application we showed him the extensive article by Edward Mellanby, Professor of Pharmacology, University of Sheffield, Sheffield, England, which was published in a report of the Privy Council, Medical Research Council, published by His Majesty's Stationery Office, London, England, in 1925, under the title of Experimental Rickets.

This article shows a great deal of research on the toxic property of wheat germ oil as affects rickets and showed clearly that wheat germ was one of the worst rachitic agents known. For example, see Pages 13, 14, 15, 16, 53, 55, 56, 57, 58 and particularly 59 and 64.

At Page 59 Mellanby said:

"In some experiments, however, it seemed that commercial wheat germ had a peculiar effect of its own. For instance, both in 803 (saponifiable substances of wheat germ) and 807 (wheat germ) the animals developed a peculiar nervous condition, the most obvious feature of which was the inability to walk or run straight. The animals seemed to lose their sense of balance and their heads moved from side to side when they attempted to walk, and finally they fell over. At a later period they were unable to stand, partly because of their loss of balance and partly because of muscular weakness. These conditions were not obviously related to the development of rickets because, as explained above, 803 was practically free from rickets and yet was the more affected of the two dogs. The other animals of this litter, even 805 (saponifiable substances oatmeal), which developed severe rickets, did not show any of the symptoms of unbalance seen in the germ-eating dogs, although 805 de-

veloped great muscular weakness. In five other cases where germ or ether or acetone extract of germ has been included in the diet of puppies notes were made to the effect
215 that the animals show lack of balancing power. There are several cases, however, where there was no such development under these or closely allied dietetic conditions. Evidence for the presence of a constituent of germ partially removed by either extraction, and capable of acting on the nervous system and especially of bring about loss of balance, is fairly strong in these experiments, but is not conclusive. If this substance is also present in other cereals, it must be in smaller amounts than in wheat germ. On the other hand, the substance in oatmeal which interferes so strongly with bone-calcification is present in smaller amounts in wheat germ. It would be interesting if it should prove that the different effects of toxic action produced by oatmeal and wheat germ, when forming large parts of diets deficient in anti-rachitic vitamin, should depend on different substances both of which are associated with the fatty acid fraction."

We think that these references are ample to make it clear that wheat germ has not been favorably regarded as a food product.

Certainly the only reference which indicates its use in any way is Hoffman and, as already pointed out, Hoffman does not deal with the product as a finished, complete and inherently palatable food product. In order to make this clear, we think it might perhaps be valuable to discuss the Hoffman reference somewhat at length.

He first frees the wheat germ from oil either with benzol or carbon tetrachloride. Then without freeing the product from the solvent he cooks the oil-free germs in order
216 to gelatinize the starch in them. As he says on Page 1, lines 34 to 38:

"It is desirable and necessary that the raw materials be thoroughly cooked in order that the mass may be in proper condition to respond to the treatment administered during the subsequent steps of the process."

He then subjects the entire mass to the action of malt digestion for the purpose of converting the starch to soluble sugar. As a result he obtains a liquor which is filtered off and subsequently concentrated to the desired consistency.

There is thus no suggestion whatsoever in Hoffman of the use of the wheat germ itself as a food product. All that he shows is the use of a converted sugar solution which includes some of the Vitamin B. The remainder of the wheat germ is discarded. This is certainly not a teaching that the whole germ, except for the oil, may be prepared into a palatable, dry food product. It does nothing to overcome the effect of the other references which we have shown which indicate the unpalatability and toxicity of wheat germ per se.

The disposition of Hoffman completely disposes of all of the references.

The broad claims relating to a palatable wheat germ product, including the new claim 16 which relates to any food product containing the wheat germ in this form, are clearly patentable.

217 The method claims and the narrower product claims, particularly those relating to solvent-free products, are additionally patentable for entirely distinct reasons.

Wheat germ is quite an absorbent material and there is considerable difficulty in freeing it from the solvent used. Applicant discovered that by using a solvent which would hydrolyze that the last traces thereof could be readily removed. He uses, therefore, carbon tetrachloride or ethylene dichloride. These materials hydrolyze to an acid under the conditions employed which assists in the removal of the last traces of the solvent. This step is not taught by the new reference Langfeldt. Neither is it taught by the reference Hoffman.

While Hoffman employs carbon tetrachloride as one of his suggested solvents, he has no suggestion about freeing the product from the solvent by distillation or otherwise.

Nor does he have any statement as to conditions under which the product would hydrolyze during the solvent-freeing process.

Claim 14, for example, in the present case calls for removing the last traces of solvent in the presence of heated moisture. There is no indication whatsoever in Hoffman of such a step.

We also wish to call attention to claim 13 which calls for a flour having the protease content substantially destroyed. The process which will preserve the Vitamin B and yet destroy the protease is described on Page 9. There
218 is no indication whatsoever in the art that it is possible to destroy one while preserving the other.

New claim 17 has been added as a method covering the particular temperatures employed for this purpose.

Respectfully submitted,

DYRENFORTH, LEE, CHRITTON & WILES
Attorneys

Chicago, Illinois
July 11, 1938

* * * * * * * * * *

219 *Affidavit*

Sir:

STATE OF ILLINOIS,
County of Cook, ss.

Ezra Levin, being first duly sworn, on oath deposes and says that he is the applicant in the above entitled application, and that he completed the invention of the above entitled application prior to February 16, 1931 by preparing in this country a finished, stable, marketable, non-toxic, and product having a high content of Vitamin B and containing palatable dry food product from whole wheat germ, the final substantially all of the constituents of the natural grain germ except the oily constituents.

Applicant prepared such product by treating the wheat germ with hot carbon tetrachloride. The oil was dissolved by the hot solvent and the solution rose to the top of the grain. This was removed and more solvent added. After substantially all of the oil had been removed from the
220 wheat germ, the remaining solvent was drained and the solvent removed from the grain.

During the process, the carbon tetrachloride hydrolized to produce an acid condition in the wheat germ.

Affiant is filing herewith as Exhibit A a photostat of an apparatus designed by applicant prior to February 16, 1931, the drawing itself being prepared by Wurster & Sanger prior to February 16, 1931, which apparatus was designed for and used for this process. Previous to the building of the apparatus shown in Exhibit A, the method was carried out on a laboratory scale, and applicant does not now have available any drawing showing the laboratory equipment.

Affiant further states that the apparatus was built by Groen & Co. and that he diligently proceeded with the building of a commercial apparatus as shown in Exhibit A and completed it after February 16, 1931 and thereafter used it in the commercial preparation of wheat germ oil. Exhibit B attached hereto is a copy of a check paid to Groen & Co. in part for said apparatus.

Affiant further states that he does not know and does not believe that the invention has been in public use or on sale in this country or patented or described in a printed publication in this or in any foreign country for more than two years prior to his application Serial No. 683,085, filed July 31, 1933, of which this application is a continuation-in-part, as to anything shown in said application, or for more than two years prior to the filing date of this application
221 as to anything disclosed in this application, which is not disclosed in said parent application; and affiant further states that he has never abandoned the invention.

EZRA LEVIN.

Subscribed and sworn to before me this 3rd day of June, A. D. 1938.

NANCY MATHEWS
Notary Public

My commission expires Feb. 11, 1942.

* * * * * * * * * *

249 Responsive to amendment of July 14, 1938.

Dietz is withdrawn as a reference.

Claims 3, 4, and 6 to 14 are again rejected on the art (other than Dietz) of record. It is not apparent that it would be unobvious to use a heated organic solvent to extract wheat germ oil. The art teaches the treatment of wheat germ. Applicant is not the discoverer of the valuable properties of the germ. Solvents are commonly employed in extracting vitamins. Langfeldt removes all of the solvent after a vitamin extraction. Hoffmann and Sherman disclose the valuable properties of the germ. It is not apparent that invention resides in omitting the malting step of Hoffmann.

In regard to the product, it appears that it differs, if at all, only in degree from the prior art products.

New claims 16 and 17 are rejected on the art for the reasons applied to the rejection of claims 3, 4, and 6 to 14. The temperatures of claim 17 are not patentable features.

This action is Final.

J. ISAACS
Acting Examiner.

* * * * * * * * * *

250 Hon. Commissioner of Patents
Washington, D. C.

Sir:

It is respectfully requested that the affidavit of Ezra Levin filed herewith be entered in the above application and be made a part of the record for the purpose of appeal.

Respectfully,
DYRENFORTH, LEE, CHRITTON & WILES
Attorneys

* * * * * * * * * *

251 *Affidavit*

Hon. Commissioner of Patents.
Washington, D. C.
Sir:

STATE OF
County of , *ss.*

Ezra Levin, being first duly sworn, on oath deposes and says that he is the applicant in the above entitled application; and that he is the President and a stockholder of the VioBin Corporation, an Illinois corporation, with offices at 160 North Wells Street, Chicago, Illinois.

Affiant further says that the said VioBin Corporation is, and has been for a number of years, processing wheat germ material in accordance with the process disclosed and claimed in the above application; that said corporation has produced thereby a product claimed in the above application and has marketed the same; that the product so produced has been widely sold in the trade, both in bulk form to producers and in the retail market, under the trade names of "VioBin", "Life of the Wheat", and "B-7"; and that said product is referred to herein as VioBin.

252 Affiant further says that VioBin has been sold as a product to be incorporated in flour, bettering the baking qualities and preventing deterioration which occurs in the use of the raw wheat germ; that VioBin has been sold for this purpose to Baur Flour Mill, St. Louis, Missouri, since the month of February, 1938; that approximately 11,200 pounds have been sold to said mill since that time; that the amount sold to said mill has on the average increased from month to month; and that during the month of January, 1939, 1,400 pounds were sold to said mill for this purpose.

Affiant further says that VioBin has been sold to the Eloise Hospital of Wayne County, Michigan, for incorporation in breads; and that during the month of December, 1938, 2,000 pounds were sold to said hospital.

Affiant further says that VioBin has been sold to the Jewel Tea Company of Barrington, Illinois, for addition to malted milk and powdered sugar; that sales to this company began on or about May 18, 1936; that since that date approximately 41,530 pounds have been sold to the Jewel Tea Company for this purpose; that throughout this whole period not a single complaint as to the spoilage of the product has been received; that there is no competitive product to which is even attributed the same stability in this field; and that to this date during the month of February, 1939, orders for 8,400 pounds of VioBin have already been received from said company.

253 Affiant further says that VioBin has been sold to be used in peanut butter to replace the Vitamin B Complex which is removed when the embryo of the peanut is extracted; that VioBin furnishes a wheat embryo in a stabilized form; that for this purpose VioBin has been sold to Miller's Peanut Product Co., 1990 Gratiat Avenue, Detroit, Michigan; and that during the month of December, 1938, 500 pounds were sold to this corporation for this purpose.

Affiant further says that VioBin Corporation has engaged in processing wheat germ in accordance with the process of the above application for the Ogilvie Flour Mills, Montreal, Quebec, Canada; that the Ogilvie Mills ship to the VioBin Corporation the raw wheat germ which is then processed and returned to said mills for packaging and distribution; that the product is sold throughout Canada in an ordinary paper container of the type used in packaging cereals; that processing for this corporation was begun on or about June 13, 1938, and has continued to this date; that an average of approximately 10,000 pounds of wheat germ per month have been processed for said Ogilvie Mills; and that during the month of January, 1939, approximately 12,600 pounds were so processed.

Affiant further says that VioBin has been sold to the Canadian Canneries of Toronto for use in place of the raw

wheat germ; that in such a use the advantages of a standard product resistant to spoilage are obtained; and that 100 pounds of VioBin were sold to said canneries in August, 1938.

254 Affiant further says that VioBin has been sold as an ingredient for pharmaceutical tablets for a source of Vitamin B Complex and minerals of wheat; and that for such purposes substantial quantities have been sold to the Vita Minerals Corporation, 3636 Beverly Boulevard, Los Angeles, California.

Affiant further says that VioBin has been approved and accepted by the Council on Foods of the American Medical Association; and that the VioBin Corporation is authorized to indicate such approval upon the containers in which VioBin is sold.

Affiant further says that VioBin has been widely sold to pharmacies and prescribed by physicians; that this is particularly true in the Chicago area which is the only district in which the VioBin Corporation has made an effort to merchandise its products through the medical profession; that, for example, VioBin is sold to Frank B. Willis & Company, distributor, 205 West Wacker Drive, Chicago, Illinois; that this company distributes and sells VioBin for this purpose; that VioBin has been sold to this company since July 21, 1937; that to this date an average of approximately one gross of packages per month have been sold to said company; that the sale has continuously increased; and that during the month of December, 1938, for example, three gross of packages were sold to this company.

Affiant further says that VioBin is packaged and sold to Park & Smith, of Detroit, Michigan, for distribution and sale under the name of "B-7"; that VioBin was first

255 sold to said firm on June 10, 1937; that the amount sold to said firm has continuously increased since that date; that approximately 7,100 pounds of VioBin have been sold to said firm; and that during the month of December, 1938, 640 pounds of VioBin were so sold.

Affiant further says that VioBin has been sold in the retail market in the form of small packages such as are used for cereals and the like; that in October, 1938, an order was received from Sears Roebuck & Company of Chicago, Illinois, for packaged VioBin sold under the trade name of "Life of the Wheat"; and that during the month of December, 1938, 24 dozen packages were sold to said company.

Affiant further says that VioBin is being sold as a food for fur bearing animals which require a stable non-rancid wheat germ; and that VioBin is used as an ingredient of a number of health food products.

Affiant further says that during the month of December, 1938, 1,026 packages of VioBin, 744 packages of "Life of the Wheat", and 640 packages of "B-7" were sold for the retail market.

Affiant further says that the uses of VioBin have been expanded progressively and continuously; that the sales of VioBin in practically every field have continuously increased; that during the month of December, 1938, 10,000 pounds of VioBin were processed and sold; and that during the same month, including the processing for the Ogilvie Flour Mills, 21,000 pounds of wheat germ were processed in accordance with the above application.

256 Affiant further says that VioBin, because of the cost of processing, is necessarily sold at a much higher price than is raw wheat germ; and that notwithstanding this high price a large portion of all VioBin sold is used by the manufacturers to replace the raw wheat germ.

EZRA LEVIN

Subscribed and sworn to before me this 9 day of Feb, 1939.

SOL SIMON
Notary Public.

Notary Public, Kings Co. No. 196 Reg. No. 313
Cert. filed in N. Y. Co. No. 452 Reg. No. 6S308
Commission Expires March 30, 1940

* * * * * * * * * * *

257 Hon. Commissioner of Patents
Washington, D. C.

Sir:

Applicant hereby appeals to the Board of Appeals from the decision of the Examiner in the matter of the above application, and more particularly from the rejection for the second time for the same reasons of Claims 3, 4, 6 to 14, 16 and 17, by action of the Primary Examiner dated August 15, 1938; the following grounds being assigned therefor:

1. The Examiner erred in rejecting Claims 3, 4, 6 to 14, 16 and 17.

2. The Examiner erred in not finding Claims 3, 4, 6 to 14, 16 and 17 patentable over the cited art.

3. The Examiner erred in rejecting claims 3, 4, 6 to 14, 16 and 17 upon Hoffman, Sherman and Langfeldt.

258 4. The Examiner erred in rejecting Claims 3, 4, 6 to 14, 16 and 17 upon Sure, Hoffman, Sherman and Smith in view of the state of the art.

5. The Examiner erred in not allowing Claims 3, 4, 6 to 14, 16 and 17.

The appeal fee of fifteen dollars is enclosed herewith.

Respectfully submitted,

DYRENFORTH, LEE, CHRITTON & WILES
Attorneys.

Chicago, Illinois
February 8, 1939.
WBG:y

* * * * * * * * * *

259 The affidavit submitted February 13, 1939, has been entered.

The affidavit has been carefully considered. However it appears to furnish evidence relating to commercial factors and to stability of the product. It is not apparent that there is any evidence which has a bearing on the rejection of the claims. The merit of the product has not been ques-

tioned. The rejection is based upon lack of patentable novelty.

The appeal will be answered in due course of business.

A. H. WINKELSTEIN,
Examiner.

L

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260 *Examiner's Statement.*

This is an appeal from the rejection of all the claims.

The claims are 3, 4, and 6 to 17, inclusive.

The claims are reproduced below:

* * * * * * * * * *

262 The art relied upon is:

Hoffman,	1,640,193,	Aug. 23, 1927
Sure,	1,869,721,	Aug. 2, 1932
Langfeldt,	1,925,489,	Sept. 5, 1933

Sherman, Chemistry of Foods, 1920, Pp. 349-350.
Sherman and Smith, The Vitamins, 1922, page 45.

This application relates to the treatment of the embryos of cereal grains to obtain a product rich in vitamin B. It is known that cereal grain germs tend to turn rancid on standing. To avoid this rancidity, which is caused by the germ oil, applicant extracts the oil from the germ.

Applicant heats the germ in an acid condition with a solvent which will extract the germ oil. The temperature is maintained between 125° F. and 160° F. The solvent containing the oil is drawn off, and residual solvent in the germ is removed by additional heat in the temperature
263 range used during the extraction. While ethylene dichloride is a preferred solvent, applicant may use any suitable petroleum hydrocarbon solvent having a relatively low boiling point. By using solvents which hydrolyze during the treatment, it is not necessary to add acid. Such solvents are various chlorine derivatives of ethane.

The Claims.

Claims 3, 6, 7, 12, 13, and 16 are drawn to the oil free germ product.

Claims 4, 8, 9, 10, 11, 14, 15, and 17 are drawn to the method.

The product claims define essentially a grain germ consisting of all the constituents of natural grain germ except the oily part. Claims 6 and 7 include process limitations describing the method of oil removal. Claim 13 describes a flour comprising the oil free germ. This claim also includes process limitations. Claim 16 is broader than claim 13. Claim 16 covers any food comprising the oil free germ.

Claim 4 covers the broad method of removing the oil with a heated solvent, and then removing the solvent.

Claim 8 depends from claim 4, and specifies a temperature range of 125° to 160° F. The claim also includes the use of a vacuum.

Claim 9 depends from claim 4. The claim specifies that the solvent is ethylene dichloride.

Claim 10 employs a class of solvents consisting of chlorine derivations of the lower molecular weight paraffins.

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Claim 11 depends from 10. This claim also employs steam to produce a partial hydrolysis.

Claim 14 employs a hydrolyzing solvent which is removed by drainage and heat.

Claim 15 adds vacuum to the process of claim 14.

Claim 17 uses a temperature range of 125° to 160° F. for treating the germ and for extracting the solvent.

The Rejection.

All the claims stand rejected as lacking invention over Hoffman, Sherman, or Sherman and Smith. Hoffman discloses the preparation of a food product of high vitamin content. The product is produced from cereal germs such as wheat germ. Hoffman first frees the germ of its oily constituents, since, as he points out:

"Without extraction of the oil from the cereal germs, it is impossible to produce a product having a nutritive value as great as that possessed by the present product."

Hoffman cooks the oil free germ and then subjects the germs to a malting action to convert the starch into soluble sugars. The extraction is carried out with benzol or carbon tetrachloride.

Applicant argues that since Hoffman malts his germs that reference is of no value. The argument overlooks the fact that Hoffman cooks and malts his germs for the express purpose of gelatinizing and saccharifying the starch. Obviously, if this formation of soluble sugars is not desired, the steps and their corresponding functions could be omitted. The removal of oil by Hoffman and by applicant is carried out essentially in the same manner. That is the crux of the present application. The avoidance of steps added by Hoffman does not indicate an inventive advance over that patent.

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Sherman discusses the nutritional values of wheat embryo. He states that there is a toxie substance in the germ, and that substance is associated with the fat. He discloses that extraction of the fat removes in great measure the toxicity of the germ.

Sherman and Smith also disclose a solvent treatment of wheat germ.

There is, then, a definite disclosure in the art of the preparation of oil-free cereal germs. That germ product reads on the product of claims 3, 6, 12, 13, and 16. The process limitations 6, 7, and 13 do not distinguish the product in any patentable sense. Such limitations are, it is submitted, improper in these product claims; these claims being additionally rejected for this reason. The matter of the removal of solvent is one of degree and not in kind. The oil extraction shown by Hoffman and Sherman would extract the harmful constituents and leave the desirable vitamin B in the germ. In this connection, it may be pointed out that carbon tetrachloride, employed by Hoffman, is a hy-

drolyzable solvent, which, under the influence of heat, would produce an acid condition.

As to the method claims, the temperature range of 125° to 160° F. is not of patentable significance. The temperature would normally be such as to avoid any destruction of desirable enzymes. The use of a low temperature is made possible by the obvious expedient of using a vacuum.

Applicant urges strenuously the complete removal of solvent from the germ following the extraction step. It is obvious that normal chemical procedure would dictate the complete removal of solvent from the germ which is intended to be taken as a food substance. At most
266 the residual solvent, if any, would differ in quantity only, but not in a patentable sense, over the amount of solvent in applicant's product. Langfeldt appears to remove all the solvent in a vitamin extraction process carried out in apparatus similar to that used by applicant.

The use of an acid condition during the extraction follows naturally from a knowledge of the labile nature of vitamin B. As pointed out by Sherman and Smith, wheat embryo contains vitamin B. They disclose that in extracing wheat germ with such solvents as ether, benzene, or acetone, the vitamin B content is not disturbed. They point out that vitamin B is destroyed by alkalies and is stable toward acid. This position is confirmed by Sure who uses an acid extracting solvent in extracting vitamin B containing materials. Sure points out that slight acidity is all that is required.

It is pointed out, moreover, that the carbon tetrachloride used by Hoffman is not only equivalent to ethylene chloride, but also hydrolyzes to produce the acid condition set forth in the process claims.

Applicant has submitted a number of affidavits which do not appear to furnish any evidence bearing on the merits of the claims. The affidavits relate essentially to commercial factors and to the merits of the product. The merit of the product has not been questioned.

It is the Examiner's considered opinion that the claims do not set forth any unobvious procedure in view of the prior art. The product differs only in degree, if at all, from prior art products.

Respectfully submitted,

A. H. WINKELSTEIN,
Examiner, Div. 63.

* * * * * * * * * * *

282 Before the Board of Appeals

Ex parte Ezra Levin

Application for patent filed August 6, 1936, Serial No. 94,709. Method of Treating Cereal Grain Germ.

Messrs. Dyrenforth, Lee, Chritton & Wiles for applicant.

This is an appeal from the decision of the examiner finally rejecting claims 3, 4 and 6 to 17 inclusive.

The following claims are illustrative:

3. A finished stable, marketable, non-toxic and palatable dry food product with a high content of vitamin B, consisting of all the constituents of natural grain germ except the oily constituents thereof and with such constituents unimpaired and in the same condition as in the natural germ.

4. The process of making a finished stable, marketable, non-toxic and palatable dry food product, consisting in extracting from natural grain germ all of the oil with a heated solvent therefor at a temperature and acidity to maintain the vitamin B content unimpaired, and subsequently completely removing the solvent from the germ.

The references relied upon are:

283	Hoffman	1,640,193	August 23, 1927
	Sure	1,869,721	August 2, 1932
	Langfeldt	1,925,489	Sept. 5, 1933

Sherman, Chemistry of Foods, 1920, Pp. 349-350
Sherman and Smith, The Vitamins, 1922, page 45.

The examiner has set forth the invention as follows in his statement:

"This application relates to the treatment of the embryos of cereal grains to obtain a product rich in vitamin B. It is known that cereal grain germs tend to turn rancid on standing. To avoid this rancidity, which is caused by the germ oil, applicant extracts the oil from the germ.

Applicant heats the germ in an acid condition with a solvent which will extract the germ oil. The temperature is maintained between 125°F and 160°F. The solvent containing the oil is drawn off, and residual solvent in the germ is removed by additional heat in the temperature range used during the extraction. While ethylene dichloride is a preferred solvent, applicant may use any suitable petroleum hydrocarbon solvent having a relatively low boiling point. By using solvents which hydrolize during the treatment, it is not necessary to add acid. Such solvents are various chlorine derivatives of ethane."

All of the claims have been rejected as being unpatentable over the Hoffman, Sherman and Sherman et al patents. Claims 3, 6, 7, 12, 13 and 16 are drawn to cover a product. These claims will be considered first. They cover a food product comprising a natural grain germ having present in it and unimpaired all its natural constituents except the oily constituents which have been removed. The product is defined as being non-toxic, stable and palatable in some claims. Some claims specify that the vitamin B potency is substantially the same as in the original germ.

The examiner relies mainly on the Hoffman patent in rejecting the claims. It is clear that in this patent cereal germs such as wheat germs, are treated to produce a
284 product having a high water soluble B vitamin content. The first part of the process consists in free-

ing the wheat germs from their vegetable oil by subjecting them to an extracting agent such as benzol or carbon tetrachloride. The patent states that the cereal germs are freed from their oily content. The process so far is comparable with that of applicant's as applicant employs an extracting agent of the same character as carbon tetrachloride. Several solvents that may be used are mentioned by applicant on page 10 of his specification. It does not seem to be seriously contended that the solvent used by Hoffman does not hydrolyze in the same manner as in applicant's process so as to maintain the solution in an acid condition during extraction. After the oil has been dissolved Hoffman treats the oil free germs further to obtain his final product. The examiner relies on the intermediate product obtained by Hoffman after the oil has been dissolved to render the claims unpatentable. He recognizes that the solvent is still present in the wheat germs at this stage but states that no invention would be involved in removing the same so as to obtain, if desired, a dry palatable product instead of going through the remaining steps of Hoffman to obtain a different product.

In answer to this position, applicant argues that it would be very difficult to completely remove the solvent to obtain a palatable product. He argues that even if it be tried
285 it would require invention to do so. He argues that Hoffman does not even suggest that this should be done and discloses no way to do it.

Many of the product claims before us define over the intermediate product of Hoffman mainly by the recitation that the product is oil free and is palatable. We do not think that any invention is involved in removing from the wheat germs the solvent which has dissolved the oil if a dry product is desired. Whether it is difficult to remove the last traces of the solvent is not known but it is quite a common practice to subject materials to the action of steam to remove the last traces of solvents therein. At any rate, to render a product palatable may not require the removal of

the very last traces of a solvent as a product having some solvent in it may be palatable to some persons although not to others. We do not think that this limitation is sufficient to define over the Hoffman patent. We think claims 3, 6, 12 and 16 are not patentable.

The product claims 7 and 13 recite that the product has its protease substantially destroyed. On page 9 of the specification it is stated that the process must be carried out under certain specific conditions in order to destroy the protease. It is also stated that the protease must be destroyed in order to render it suitable as a flour. The references do not teach anything in this respect and we think therefore that these two claims contain novel subject matter. The rejection of claims 7 and 13 on the art will not be affirmed. However, the examiner has rejected these
286 claims on the ground that they improperly define the product because they include a process step, namely, "freed from oil by solvent extraction". We think this ground of rejection is well taken and will affirm the rejection of these claims on this ground. Applicant has proposed a claim on page 11 of his brief which covers the same subject matter as claim 7 but omits the process step. If applicant will submit this claim by a formal amendment we recommend its entry and allowance thereof.

The process claims 4, 8, 9, 10, 11, 14, 15 and 17 stand rejected on Hoffman and the Sherman and Sherman et al references. It is clear that Sherman et al discloses that vitamin B in a solution extracted from wheat germs by alcohol shows marked stability toward hydrochloric acid but is rapidly destroyed by alkalies. This teaching is believed to be sufficient to indicate to any one skilled in the art that the extraction of the oil from the wheat germ should be carried out under acid condition so as to not destroy the vitamin B. Such acid condition can be obtained by adding acid or by using a solvent which will hydrolyze and thus maintain the acid condition. Therefore, we think that claims 4, 9, 10, 14 and 15 are regarded unpatentable over Hoffman in view of Sherman et al. It would not require any invention to

carry out the extraction in Hoffman under acid conditions and to remove the solvent from the intermediate product of the solvent to obtain a dry product. The recitation
287 of certain temperature limitations in claim 8 is not regarded sufficient to render this claim patentable. As to claim 17 there is included the step—

* * * "removing the last traces of solvent in the presence of heated moisture at a temperature between 125°F and 160°F whereby the protease in the grain is destroyed."

This step is not disclosed by the art and we think this claim is patentable.

We have carefully considered applicant's brief and the various points emphasized but we think that most of the claims before us are so broad that they fail to define over the art cited.

The decision of the examiner is affirmed as to claims 3, 4, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15 and 16 is affirmed and is reversed as to claim 17.

Board of Appeals

HENRY VAN ORSDALE
Assistant Commissioner

T. P. EDINBURG
Examiner-in-Chief

J. W. CLIFT
Examiner-in-Chief

Sept. 25, 1939

Messrs. Dyrenforth, Lee, Chritton & Wiles,
850 Munsey Bldg.,
Washington, D. C.

* * * * * * * * * *

290 *Ex parte* Ezra Levin,
Serial No. 94,709,
Filed August 6, 1936,
Method of Treating Cereal Grain Germ.

Notice of Civil Action Under Section 4915 R. S.

A civil action under Section 4915 R. S., entitled Ezra Levin v. Conway P. Coe, Commissioner of Patents, Civil Action No. 5342, involving this application, was filed on January 6, 1940, in the District Court of the United States for the District of Columbia.

W. W. COCHRAN
Attorney for the Commissioner.

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Patented Aug. 23, 1927. 1,640,193 84

UNITED STATES PATENT OFFICE.

CHARLES HOFFMAN, OF TUCKAHOE, NEW YORK, ASSIGNOR TO WARD BAKING COMPANY, OF NEW YORK, N. Y., A CORPORATION OF NEW YORK.

PROCESS FOR MAKING FOOD PRODUCTS.

No Drawing. Application filed August 20, 1924. Serial No. 733,110.

The present invention relates primarily to a process for the manufacture of a food product having a notably high vitamine content. It has to do particularly with an improvement and further development over the process disclosed in the patent of Charles Hoffman, Harris Davett Grigsby and Nathan Minton Cregor, No. 1,541,263, granted June 9, 1925, to the assignee of the present application.

In its preferred form, the product consists of a dry powder of yellowish brown color having a high content of soluble sugars and characterized by a sweet malty taste. The material also has a high content of protein and mineral salts and additionally serves as a carrier for a large amount of vitamines, notably the water soluble B vitamine. A particular merit of the manufacture is that the product is produced from cereal germs, such as for instance, wheat germs, corn or maize germs and rice germs. Cereal germs constitute a by-product of milling processes and are readily obtainable at low cost.

In carrying out the process, the cereal germs are first freed from their oily content. Without extraction of the oil from the cereal germs, it is impossible to produce a product having a nutritive value as great as that possessed by the present product. The oil free germs are then cooked in order to gelatinize the starch contained in them. It is desirable and necessary that the raw materials be thoroughly cooked in order that the mass may be in proper condition to respond to the treatment administered during the subsequent steps of the process. The mass containing the gelatinized starch is then subjected to the action of malt diastase for the purpose of saccharifying the starch and converting it into soluble sugars. The resulting liquor is filtered off and is subsequently concentrated to the desired consistency. The process just outlined corresponds to that described in the above mentioned copending application.

In the carrying out of the process, however, much difficulty was encountered in securing a complete digestion of the cooked, oil-free cereal germs so that the resulting product was free from starches. The present invention contemplates the elimination of this difficulty, and it is a principal object of this invention to provide a process of the above general nature for the manufacture of a palatable food product of high nutritive value such that the digestion of the starches by the malt takes place rapidly and completely and without deleterious effect upon the flavor or other characteristics of the ultimate product.

The digestion of the oil free cereal germs with malt proceeds slowly and, in fact, it was found practically impossible to secure such a complete digestion of the mass as would render the product starch free after the malting operation. Periods of digestion ranging up to as much as two hours were insufficient to accomplish such a thorough conversion of the starch that the malted mixture would appear to be starch free as indicated by the starch iodine test. Similarly the use of larger quantities of malt, differences in the time and temperatures of cooking, differences in temperatures of mashing and the like, were of but little avail in securing a better and more complete conversion of the starches during the malting operation.

The present invention contemplates the rapid and complete digestion of the cooked raw materials by conducting the malting operation under a properly controlled acid reaction. When the malt digestion of the cooked product was made with water of neutral or but slightly alkaline reaction, the filtered malted liquor and the ultimate product exhibited an acidity expressed as pH of from 5.9 to 6.0. The pH value of the solution was measured by the standard potentiometer method based on the electrical conductivity of the substance. As the result of experimentation, I found that as the acidity was increased, within certain limits, the action of the malt upon starches of the batch was favored and complete digestion of the contained starches was attained in a reduced time. I also found that when the acidity of the batch exceeded cer-

tain limits, the ultimate product was characterized by a decidedly disagreeable bitter taste. The present invention therefore contemplates the addition to the cooked oil free cereal germs of acid in such amount as to permit rapid digestion of the starches by the malt and at the same time to preserve a palatable flavor in the ultimate product.

I have found that for the production of the best product, the amount of acid added should be sufficient to give to the ultimate product a pH value of from 5.4 to 5.7. Larger amounts of acid can be added with beneficial results in the malt digestion step, but the use of such amounts of acid ordinarily introduces a bitter taste in the ultimate product. Acid may be added in such quantity that the pH value of the ultimate product will be as low as 5.1 without rendering the product definitely unpalatable, but I prefer to maintain the acidity of the ultimate product at a pH value of between 5.4 and 5.7. A high concentration of acid apparently has the effect of extracting and communicating to the ultimate product certain bitter principles contained in the raw materials or results in a breaking down of some of the nitrogenous constituents of the raw material to form unpalatable compounds. The fact is that with too high an acid concentration, the ultimate product has a disagreeable bitter taste.

Neutralization of excess amounts of acid used in the process, while serving to bring the pH value of the ultimate product within the desired limits, nevertheless do not serve to render the product palatable. The taste of the ultimate product is substantially the same as that observed when similar excess quantities of acids were used and were present in the ultimate product.

The effect of the presence of acid during the malt digestion of the cooked oil-free cereal germs may be well illustrated experimentally. For instance four batches, consisting each of 200 parts of oil-free wheat germ, 30 parts malt, and 1500 parts water were subjected to processes identical with the exception that for batch 1, no acid was added during the malting step; for batch 2, .36 parts of acid were added during the malting operation; for batch 3, .72 parts of acid were added, and for batch 4, 1.08 parts of acid were added. The acid used was concentrated sulfuric acid. For batches 3 and 4, the excess acidity was neutralized with calcium carbonate. The ultimate product showed for batch 1 a pH value of 5.9, for batch 2 a pH value of 5.6, for batch 3 a pH value of 5.6 and for batch 4 a pH value of 5.4. Batch 1 was subjected to a malt digestion for a period of two hours and even after this extended period of digestion, small amounts of starch still remained unconverted by the malt diastase. Batch 4 was starch free after only 15 minutes of malt digestion and batches 2 and 3 were starch free after a digestion of slightly more than 15 minutes. The resulting products showed considerable differences in flavor. Batches 1 and 2 were of substantially identical flavor and were entirely palatable and pleasing to the taste. Batches 3 and 4 both exhibited a sharp and slightly bitter taste. My experimentation demonstrated that the best product is obtained when the amount of acid added serves to give the ultimate product a pH value of between 5.4 and 5.7, no basic neutralizing agent being added. With such a quantity of acid, the length of time required for complete digestion of the starches by the malt is short and requires but from 15 to 20 minutes.

In carrying out my improved process, cereal germs are first freed from their vegetable oil by a suitable extracting agent, such as benzol or carbon tetrachloride. Either of these extraction agents is available commercially and may be used economically because of low cost, efficiency in extracting the oily matter and ready regeneration. The oil free germs are then cooked preferably under a pressure of about 15 pounds per square inch gauge for from 15 to 30 minutes. I have found that better results are obtained by cooking the raw materials at relatively high pressure and temperature for a relatively short length of time rather than conducting the cooking operation at atmospheric pressure and temperature for a greater length of time. The cooked mash is then cooled to 133° F. at which point the requisite amount of acid is added. In the case of sulfuric acid for instance, an amount of acid in the neighborhood of .36 parts of concentrated sulfuric acid to 200 parts of oil free germ is suitable. After the addition of acid the material is malted preferably by the addition of a malt infusion prepared by digesting 20 parts of malt and 250 parts of water at 82° F. for 15 minutes, then at 100° F. for 15 minutes and then at 133° F. until needed for addition to the batch. The malted mash is then digested for a proper length of time, say 15 to 20 minutes, until the starch iodine test shows the material to be free from starch. The liquor is then filtered off and concentrated as desired. The product is preferably evaporated to dryness so that the ultimate product is a solid in the form of a powder.

The process is preferably supplemented by a second malt digestion step, the second malting operation taking place either just before filtration of the mash, immediately after filtration of the mash or after concentration of the liquor to syrupy consistency. The process employing the double malt digestion is fully disclosed in the copending application of Nathan Minton Cregor for

"process for making food product" filed concurrently herewith. No claim is made in this application to the process employing the double malting feature except when such process includes the control of the acidity during the malt digestion step as described herein. The present invention does contemplate a process employing the double malting step wherein the acidity of the mash during the malting operation is controlled after the fashion described in this application.

The character of the water used in the cooking and malt digestion operations has an effect upon the quantity of acid which must be added. With water which for instance is strongly alkaline, it is necessary to add an amount of acid greater than that above specified for the reason that an excess amount of acid is required in order that the alkalinity of the water may be neutralized and at the same time a sufficient acid concentration obtained to permit rapid digestion of the starches by the malt.

The present invention therefore contemplates a process which includes addition to the material under treatment of an amount of acid such that the malt digestion of the raw material will require a substantially shorter time and such that no disadvantageous effect in the ultimate product will be produced.

I claim:

1. The method of producing from cereal germs a food product rich in water soluble B vitamine which comprises extracting the oil from the said germs, cooking the raw material to gelatinize the starch contained therein, acidifying the said cooked material and malting said material to effect saccharification thereof.

2. The method of producing from cereal germs a food product rich in water soluble B vitamine, which comprises extracting oil from the said germs, cooking the raw material to gelatinize the starch contained therein and malting said material in the presence of acid in such amount as to permit rapid digestion of the starch and at the same time preserve a palatable flavor in the ultimate product.

3. The method of producing from cereal germs a food product rich in water soluble B vitamine, which comprises malting the raw material in the presence of acid in such amount as to permit rapid digestion of the starch and at the same time preserve a palatable flavor in the ultimate product.

4. The method of producing from cereal germs a food product rich in water soluble B vitamine which comprises malting said material in the presence of acid sufficient in amount to permit complete digestion of the starch in from 15 to 30 minutes and at the same time to avoid extraction of bitter principles from the raw material with the production of a consequent disagreeable flavor in the ultimate product.

5. The method of producing from cereal germs a food product rich in water soluble B vitamine which comprises malting cereal germs under an acid reaction, such that a rapid digestion of the contained starches is produced and a palatable flavor in the ultimate product is preserved.

6. The method of producing from cereal germs a food product rich in water soluble B vitamine, which comprises malting oil free cereal germs under an acid reaction such that the ultimate product is of acidity represented by a pH value of from 5.4 to 5.7.

7. The method of producing from cereal germs a food product rich in water soluble B vitamine, which comprises extracting the oil from said germs, cooking the raw material to gelatinize the starch contained therein, and malting said material in the presence of an amount of acid such that the ultimate product exhibits an acidity represented by a pH value of from 5.4 to 5.7.

8. The method of producing from cereal germs a food product rich in water soluble B vitamine, which comprises extracting the oil from said germs, cooking the material to gelatinize the starch contained therein, acidifying the said cooked material, malting said acidified material and filtering and concentrating to dryness the resulting liquor, the amount of acid added to said material being such as to produce in the ultimate product an acidity represented by a pH value of from 5.4 to 5.7.

9. The method of producing from cereal germs a food product rich in water soluble B vitamine which comprises extracting the oil from said germs, cooking the raw material to gelatinize the starch contained therein, acidifying the said cooked material, malting the said acidified material and filtering and concentrating to dryness the resulting liquor, the amount of acid added to said material being such as to produce a rapid digestion of the starches by the malt and at the same time preserve a palatable flavor in the ultimate product.

10. The method of producing from cereal germs a food product rich in water soluble B vitamine which comprises extracting the oil from the said germs, cooking the raw material to gelatinize the starch contained therein, acidifying the said cooked material and malting the said acidified material to effect saccharification thereof, the amount of acid added being sufficient to neutralize the alkalinity of the water used in the process and to further acidify the material under treatment to thereby permit rapid digestion of the starches by the malt and at the same time to preserve a palatable flavor in the ultimate product.

Patented Sept. 5, 1933 1,925,489

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UNITED STATES PATENT OFFICE

1,925,489

METHOD FOR THE EXTRACTION OF EMULSIFIED, SUSPENDED, OR DISSOLVED SUBSTANCES FROM LIQUIDS BY MEANS OF VOLATILE SOLVENTS

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Application July 29, 1930. Serial No. 471,578

2 Claims. (Cl. 167—81)

This invention relates to the manufacture of vitamine preparation and has for its object a method of treating liquids containing vitamines in a dissolved, suspended or emulsified condition with volatile solvents for the purpose of extracting the vitamines.

An important characteristic feature of the invention consists therein that the volatile solvent is introduced into the liquid to be treated in the form of vapour which is caused to be condensed in the liquid in the form of exceedingly small drops.

The initial material from which the vitamines are to be recovered is preferably subjected to a special preparatory treatment previously to the treatment with volatile solvent. Thus, if the initial material is in a solid condition, it will have to be dissolved or disintegrated and converted into an emulsion or suspension in a suitable liquid.

If the raw material is an oil or fat, for example fish liver oil, a suitable preparatory treatment will usually consist in a saponification treatment to convert the fat into a soap, which is then subjected to the extraction treatment in a dissolved or suspended condition.

According to the nature of the liquid to be treated and of the volatile extraction agent in question, the vapours of the extraction agent will be introduced at the bottom of the vessel, containing the liquid to be treated, or at the surface of said liquid or also at points between bottom and surface.

In case of the liquid to be treated having a higher specific gravity than the condensed solvent, it will be suitable to introduce the vapours at or near to the bottom of the extraction vessel, while in cases where the conditions are the reverse, it will be preferred to introduce the vapours at a higher point in the liquid, for example immediately below the surface of the liquid.

During the treatment with the solvent vapours, the liquid is maintained at a temperature below the boiling point of the solvent, so that the vapours will be condensed in the liquid.

When the vapour supply is arranged as above indicated, the condensed extraction agent will move through the liquid in a direction from the point of supply as a consequence of the difference in specific gravity between liquid and extraction agent, and while thus moving through the liquid, it will take up soluble substances from the liquid.

The resulting solution can be brought to flow continuously or intermittently from the extraction vessel, to be further treated for separating the dissolved substances.—The extraction agent separated from dissolved material by distillation, absorption or by other means may anew be introduced into an extraction vessel in the form of vapour.

The vapours of extraction agent are introduced into the liquid to be treated in a finely divided form, for example by being forced into the liquid through a wall of porous material or provided with fine holes. The vapours will then become condensed as very small drops, so that the extraction agent is thoroughly distributed in the liquid.

The required cooling of the liquid may be brought about by cooling means of known character, for example by passing a cooling fluid through inserted coils or other cooling channels or vessels or through cooling jackets, surrounding the extraction vessel. It is preferred to so arrange the cooling means as to allow of producing different degrees of cooling at different levels or (and) at different places in the extraction vessel, because this will besides having other advantageous effects promote the circulation and mixing of liquid and condensed extraction agent. Powerful cooling will in most instances be of advantage. Thus it will frequently be desirable to cool the liquid so strongly as to cause the liquid at the walls of the cooling members to attain a temperature near the freezing point of the liquid.

An apparatus constructed and operating in accordance with the invention is diagrammatically illustrated in the accompanying drawing. In the illustrated example, 1 and 2 are lixiviation vessels for use in cases where the volatile lixiviation liquid is lighter than the liquid to be lixiviated. 3 and 4 are lixiviation vessels for use in cases where the lixiviation agent is heavier than the treated liquid. 5,5, 6,6, 7,7, 8,8, are jackets for circulation of cooling fluid. 9 is a vessel for volatile lixiviation agent, placed in a water bath 10.

Vapours of lixiviation agent passes from the vessel 9 through the pipes 11, 12 to the lixiviation vessels 1, 2, 3 and 4. In the vessels 1 and 2 the pipes 11 open at the bottom of the vessels below a perforated partition 13. In the vessels 3 and 4 the pipes end in a perforated nozzle or spray rose 14 at the top of the vessels, 15 and 16 are pipes through which condensed volatile solvent with dissolved matter flows back into the vessel 9 at the bottom of this latter. 20 is a reflux condenser, which can be brought in communication with the top of the lixiviating vessels 1, 2, 3, 4. 22 is a funnel for charging the lixiviating vessels with liquid to be extracted. 21 is a fun-

nel through which volatile solvent is introduced into the vessel 9.

Example.—Cod liver oil is saponified with an alcoholic solution of caustic soda. The resulting solution of soaps is introduced into the lixiviating vessel (1, 2) and vapours of benzol is introduced into the solution through the porous partition. The volatile solvent condenses in the form of fine drops forming an emulsion with the soap solution. The condensed drops of solvent ascend slowly and accumulate at the top of the vessel, from where it flows back into the heated solvent reservoir.

The method and apparatus above described can be used in connection with materials of any kind, from which vitamines or other constituents are to be lixiviated by means of a volatile solvent. The solvent to be used will of course be varied in accordance with the character of the material and the conditions of operation in each instance.

We claim:

1. The method of treating solutions of a water-soluble soap containing vitamines, to separate the vitamines by means of a volatile vitamine solvent which is incompletely soluble in and has a specific gravity different from that of the said solution, which comprises vaporizing said solvent and passing the vapour in the form of fine streams into a body of said solution, maintaining the temperature of the solution considerably below the condensation point of the vapour so as to effect rapid condensation of the streams of vapour, allowing the condensed particles of solvent to pass a considerable distance through the remaining solution and then separating and collecting the solvent from the treated liquid.

2. The method of extracting vitamines from solutions of a water soluble soap containing vitamines which comprises, vaporizing a volatile vitamine solvent which is incompletely soluble in and has a specific gravity different from said solution, passing fine streams of said vapors into a body of said solution, condensing the vapor in said solution, passing the condensed particles of solvent through said remaining solution, and separating and collecting the solvent from the said solution.

EINAR LANGFELDT.
RAGNVALD HELLERUD.

Patented Aug. 2, 1932 1,869,721

UNITED STATES PATENT OFFICE

BARNETT SURE, OF FAYETTEVILLE, ARKANSAS

EXTRACTION AND CONCENTRATION OF VITAMINS

No Drawing. Application filed January 15, 1930. Serial No. 421,074.

The present invention relates to vitamins, having particular reference to a process of extracting certain vitamins from materials containing them.

Vitamins are substances only recently recognized as vital dietary components in nutrition. They occur in natural food products to a large extent, but little is known of the nature of them. A number of specifically different vitamins are referred to in various studies on them, and they are named and classified by letters, such vitamin A, vitamin B, etc. The chemical composition of vitamins is not known, but certain properties of them have been determined, and they are classified to some extent by their properties, such as fat-soluble vitamins, and water-soluble vitamins. It is now recognized that such diseases as beriberi, rickets and pellagra are caused by a marked deficiency of vitamins B, D and G, respectively. Many nutritional disturbances, characterized by lack of appetite, underweight, susceptibility to colds, etc., have been demonstrated to result from inadequite supplies rather than from total deficiency of vitamins. They are not all stable substances, and it is known that some of them are destroyed by certain chemicals, or by other conditions, such as heating. Cooking of certain foods in ordinary usage is known to lessen or totally destroy some vitamins of the uncooked food.

Vitamins were first recognized in natural foodstuffs and other materials of natural origin, both vegetable and animal. The embryos of cereals; leafy vegetables, such as spinach and lettuce; egg yolk; and glandular organs, such as liver and kidney are rich sources of vitamins found in natural foods. More recent work has demonstrated that cereals and other materials may be exposed to ultra-violet light and other radiations with the resulting production of a substance having the properties of a vitamin.

The present invention relates to a process which is specially adapted for the extraction of vitamin B from materials containing it. The process may be carried out in such a way that it is also specially adapted for extracting vitamin G from materials containing that vitamin, and the process is therefore capable of extracting both vitamin B and vitamin G from materials containing both. The invention is, of course, not limited to use of the process for natural products, for it is equally well adapted to use with irradiated products or extracts which contain these vitamins as a product of irradiation, or to other products in which the presence of such vitamins may be artificially induced.

Cod liver oil is one source of vitamins A and D. Oranges are one source of vitamin C. Rice polishings, wheat embryo, yeast, etc., are among the sources of vitamin B and also of vitamin G, both of which are considered in this process. Vitamins B and G are both water-soluble vitamins, B being less stable towards heat and in the presence of alkalis, and G being more stable towards heat. These particular vitamins are important in infant and child nutrition, and in combating diseases due to malnutrition. Both breast milk and cow's milk have been shown to be deficient in vitamin B. Pediatricians are beginning to appreciate the need of supplementing the infant's diet with vitamin B extracts. Although yeast is available as a source of vitamin B it is contra-indicated for infants because of the attendant materials which produce fermentation in the gastro-intestinal tract of infants. Wheat embryo extracts may be employed but are considered too laxative for infants, although not undesirable for adults. There is, therefore, a field for the provision of a vitamin extract which can be added to an infant's diet without interference from materials other than the vitamin.

The invention aims to provide a suitable prophylactic and therapeutic vitamin extract which can be used for infants, which is not distasteful and unpleasant of odor, which can be added to a diet of both infants, children and adults without interference from other materials contained in the extract.

A particular object of the invention is the preparation of such an extract, which may also contain vitamin G.

Another object of the invention is the preparation of an extract of vitamin B from

rice polishings, a substance which has long been used as a source of nutritious substance in infantile beriberi without harmful effect on infants. Rice polishings is a byproduct of the milling of rice, which is removed after the hulls and bran have been removed. It is the byproduct richest in vitamin B.

A further object of the invention is to concentrate the vitamin content from materials containing it, and to provide a staple product for commerce.

A further object of the invention is the preparation of a highly potent vitamin extract, and one which is relatively free from impurities such as nitrogenous matter.

Owing to the still general ignorance of the nature of vitamins and to the little known properties of these substances, it is quite impossible to point out definitely the entire limits of the invention in the manner which can be done in the more exact sciences. In this application, however, I am able to survey the path generally to be followed in preparing the extract and can give as an illustrative example of the invention specific directions of one course to follow to get the result desired. It is to be understood that the invention is not to be considered as limited to the precise disclosure that is herein made by way of illustrating the invention in order to explain it. There may be variations from the prescribed course as will naturally occur to those who are skilled in the art. Such deviations and changes will again be referred to later in the description when they will be more clearly understood.

I wish first to illustrate the invention by describing how the same may be practiced on rice polishings.

Generally the process involves extraction of the vitamin with a solvent from the base material, separation of the solvent containing the vitamin from the undissolved substance, and then removal of the solvent to leave the extracted matter as a residue. This residue is the crude extract containing other materials besides the vitamin, such as bad tasting or smelling substances which make the crude extract undesirable. The process then involves a purification by which undesirable impurities are eliminated and a useful extract or concentrate obtained.

As solvent for treating the base material I prefer to use an essentially water solvent, such as a dilute ethyl alcohol solution, I have found that a solvent which is 25% ethyl alcohol by volume and 75% water is suitable. It will, of course, be understood that other solvents may be used. A suitable solvent is one which can be readily removed by suitable means from the contained extract without damage to the extract in the removal process. The vitamins with which I am concerned are essentially water soluble and dissolve in the mixed alcohol and water as well as in water, provided a sufficient quantity is used. The alcohol aids in preventing a fermentation which might otherwise occur if water, or acidified water were used for the extraction. In using alcohol and water as described I have found that there is a tendency to destroy the vitamin B when the solvent is distilled, even at low temperaturts in a vacuum, and have found that the presence of an acid in the distillation preserves the vitamin. Consequently, I use some acid in the distillation to preserve vitamin B.

I have found that acid may be used in the extraction solvent without injuring the extraction of vitamin B. Acid tends to stabilize the vitamin B. Accordingly, when vitamin B is concerned I may add the acid either before extraction with the solvent, or before distillation of the solvent. But when I use the acid during the extraction I find there is an increase in the amount of vitamin G that may be extracted at the same time, if it is present. Therefore, when I am concerned with vitamin G alone, or both vitamins B and G, I use an acid extracting solvent.

Slight acidity is all that is required, and various acids may be used, such as acetic, hydrochloric, citric, etc. I prefer to employ any acid which may be later used in the process, and in the specific example will describe acetic acid. This acid is ordinarily considered harmless in small amounts, being the acid of vinegar having a recognized place in the diet.

As a specific example of the extraction of vitamins B and G from materials containing both of them, one may take 3600 grams of rice polishings and 20 liters of alcohol which is 25% by volume (with water) and which contains 0.5% acetic acid, or such an amount as will maintain acidity during the step to follow when the solvent is removed from the extract. The polishings and the solvent are allowed to stand with continual or intermittent agitation for about forty-eight hours at the temperature of the average living room. At the end of this period, or such shorter period as may be chosen, the polishings are allowed to settle and the liquid is decanted from the settled matter. Undissolved vitamin and that which adheres to the settlings in the solvent remaining is then removed by a washing or extraction process with a further amount of an extracting solvent. I have allowed the settlings to be again extracted with 10 liters of the same solvent in twenty-four hours with intermittent agitation. The solvent is then decanted, and the combined liquids are filtered from solid matter for recovery therefrom of a crude extract.

The combined liquids are treated to separate the solvent and the solute, as by distillation, at a temperature, and otherwise under conditions, which do not destroy the vitamin content. I have found that the alcohol and

water may be distilled off at lowered pressures without destroying the vitamin. I may use a vacuum of 50 mm. to 75 mm. pressure of mercury and a temperature of from 50° C. to 60° C. This is continued until a viscous residue is formed. The distillation may be stopped where the residue has a specific gravity of from 1.42 to 1.47.

The crude extract obtained by the above described procedure is a dark brown, gummy mass which is both acid and bitter in taste. This crude mass is an acid extract or concentrate of vitamins B and G. There will be obtained from the initial quantity of rice polishings, about 720 grams of crude extract, or about 20% yield. The extract may, of course, be used, but its taste and its impurities mitigate against desirability of using the same.

I next provide a step of purification to remove the outstanding impurities from the vitamins. Bitterness and acidity are important to remove. Color also may be lessened in the treatment which is employed. The method in general is to dissolve or suspend the whole mass in a solvent therefor, and then to dilute the mass with a diluent which forms a mixed solvent in which one or more of the impurities is insoluble and in which the vitamin content desired is soluble. By this procedure the impurities may be removed mechanically after their precipitation in and by the mixed solvent.

To the crude extract I add a solvent compatible with the next solvent to be used as a diluent. To 100 grams of crude extract I add 500 cc. of glacial acetic acid. I prefer the acid which is used in the extraction to be the same as the acid used here in purification, when such is used, for the reason that but one acid then becomes associated with the product. The suspension is accomplished at room temperature and complete mixture is made so that all the vitamin content is subjected to action of the solvent. As a precipitant, I use anhydrous acetone. This may be produced by extracting the water from a water-containing acetone in any known manner, such for example as by treatment with anhydrous sodium sulphate crystals. I use 3600 cc. of the acetone, adding it gradually with stirring to the glacial acetic acid solution of the extract. A granular precipitate occurs and rapidly settles to the bottom of the vessel. It may be separated in as soon as five minutes. The granular precipitate occurs and rapidly settles to the bottom of the vessel. It may be separated from the solution and then washed with 3600 cc. of the same character of acetone in order to remove adherent or included vitamin which can be dissolved in the acetone. The two acetone portions, one containing substantially all the acetic acid, are combined and treated for the separation of vitamin extract therefrom.

The separation of the vitamin from the solvent may be effected by a distillation under conditions which do not injure the vitamin. A low temperature distillation is suitable and may be conducted as follows: The solvent and contents are distilled in vacuo, first to remove acetone portions, and later to remove acetic acid portions. The acetone, having a considerably lower boiling point than acetic acid, may be distilled off under a vacuum of 200 mm. to 250 mm. of mercury pressure at a temperature of from 30° C. to 40° C. Two and one-half to three hours may be consumed in this operation without destroying the vitamin content. Next, the vacuum is increased and the temperature raised to remove acetic acid. This may be done at a temperature of from 40° C. to 70° C. at a mercury pressure of from 50 mm. to 75 mm. Three hours may be consumed in this operation without destroying the vitamin content. The residue is a viscous product and may contain from 10 per cent to 15 per cent acetic acid which is best not removed in the distillation. To push the distillation too hard might destroy the vitamin content. Acid exerts a preservative influence on the material, and the residue strongly holds the acid to itself.

By the above treatment the bitter taste and other impurities have been removed, but it will be noted that there is still acid remaining as an impurity. This may be reduced in part by addition of suitable alkali to neutralize the acid. But I prefer to remove it by a further evaporation treatment. To the residue remaining from the 100 grams of crude extract I add 100 cc. of distilled water, which readily dissolves the mass. The resulting liquid is then spread out in a thin film, say 2 mm. deep, and allowed to evaporate at normal temperatures of the room and at normal pressures. Thus the acid is dissipated along with the water with which it has been diluted, with the aid of a current of air, or otherwise. A residue of about 36 to 40 grams is obtained which is an amber-colored viscous liquid. It may have from 1.5 per cent to 2 per cent acidity but this is an aid in preserving the material as a more stable product. It is very potent in antineuritic, growth-promoting antiberiberi vitamin B, and contains appreciable amounts of the stable antipellagric vitamin G.

In as for as a chemical analysis may indicate the nature of the product above described, the final product may be said to contain, by a showing of analysis, the following constituents:

	Per cent
Moisture	32.0
Acetic acid	1.8
Reducing sugars as invert sugar	42.6
Ash	5.7
Fat (ether extracted)	0.3
Total nitrogen	1.1

The residual acidity is sufficient at least to keep the product stable in a refrigerator for three months. This is not unsuitable for commercial usage. I do not contemplate that the acetic acid be the only preservative for the product. Merely because it is present as a beneficial incident to its manufacture, I may follow the known practices of the art in preserving foods and such nutritional extracts. I may use as a mild preservative one-tenth of one per cent benzoate of soda. This has been effectively used for years in preserving the extracts heretofore known in the Philippines and made from rice polishings. The low nitrogen in the product may prevent or minimize a skin rash, produced by some high nitrogen products in infant's diet.

The potency of the product may, of course, vary under varying conditions of operation and of modifications of the procedure above specifically outlined. In using rice polishings and the materials and proportions herein given I have obtained a very potent product.

My extract was found to be five hundred (500) times as potent as cow's milk in vitamin B and one hundred fifty (150) times as potent as cow's milk in vitamin G. One teaspoonful of my product contains as much vitamin B as is found in 5 to 5.5 quarts of cow's milk.

A vitamin extract made by my invention may be used either as a concentrate, or it can be impregnated in various infant foods, such as dextro-maltose, lactic acid milk, various dried milks approximating the composition of breast milk, or any other kinds of foods. It is also understood that my vitamin product may be used for growing children, and adults, in cases of malnutrition, lack of appetite, and in diseases associated with loss of appetite, such as tuberculosis and pernicious anemia. I may mention in particular that the vitamin extract which I obtain from rice polishings contains a considerable amount of vitamin G along with vitamin B, and such a double extract or concentrate is beneficial in the early months of artificial feeding when the dilutions of milk are great.

It will be readily understood by those who are skilled in the art that a change in the raw material used as a source for the vitamin or vitamins may call for changes in the chemical reagents and the proportions, and conditions of operation. Also a change in the quantity of material treated in one operation, and a change of equipment used from the small scale to large scale work, will lead to economies in materials and in time, such as cannot be readily introduced nor here described for smaller batches. The character of the acid and the degree of acidity in the extraction are subject to variation. The strength of the extracting solvent may be changed, as there is nothing to indicate that it is limited to exactly 25 per cent of alcohol by volume with water. The consistency of the crude extract which is obtained may likewise be varied. In the same manner there may be changes in the quantities of solvent and precipitant used for the purification process, and the conditions under which these materials are distilled so long as the precautions against injury or destruction of the vitamin are observed. My viscous vitamin product may be dehydrated by the Spray or Roller processes, during which operations residual acidity may be dissipated. It is now known that liquids may be very rapidly evaporated at temperatures which in ordinary distillation would destroy certain products, by spraying a mist of the liquid into chambers that are heated with air currents in a vacuum. The material in such processes is so quickly passed through the heat that there is little or no change effected, comparable to that which takes place at the same temperature under longer exposure. By use of these methods a powdered product may be produced, comparable to dried milk powders, dried blood, etc., and different conditions maintained which depart from those here set forth.

Owing to the still incomplete knowledge about vitamins and their reactions, such changes in my process must be effected with caution. Gradual departure from the specified conditions of the exemplary process may, of course, be made, the effect of which may be readily determined by measuring the potency of the product resulting from this process with such variations.

Although I have illustrated my invention by describing my process specifically as I have carried it out to extract both vitamin B and G from rice polishings, it will be understood that the invention is capable of application to extract similar vitamins from other substances such as wheat embryo for example.

I do not intend the invention to be limited to the precise disclosure but aim to include such modifications and changes, and such equivalent materials as fall within the scope of the appended claims defining the invention.

The present application is a continuation in part for my former application, Serial No. 354,175, filed April 10th, 1929.

I claim:

1. The method of purifying a crude extract of vitamin B which comprises subjecting the crude extract to the solvent action of glacial acetic acid, diluting the mass with anhydrous acetone in quantity to form a mixed solvent in which the vitamin is soluble and in which an impurity is insoluble, separating the impurity from the mixed solvent, and distilling the mixed solvent from

the solute at a reduced pressure under substantially 70° C.

2. The method of purifying a crude extract of vitamins B and G which comprises subjecting the crude extract to the solvent action of glacial acetic acid, diluting the mass with anhydrous acetone in the proportion of about 36 parts of the acetone to about 5 parts of the glacial acetic acid, separating solid matter from the resulting mass, and from the resulting liquid distilling acetone and acetic acid at a reduced pressure and at a temperature not over substantially 70° C.

3. The method of concentrating vitamin B from material containing the same which comprises subjecting the material to extraction by an alcoholic water mixture, separating solids from liquid, distilling alcohol and water from the liquid in the presence of an acid under reduced pressure at a temperature not over substantially 70° C., suspending the residue in glacial acetic acid to dissolve the vitamin content, adding to the resulting mass a quantity of acetone until a soluble impurity is rendered insoluble in the resulting mixed solvent, separating the impurity from the liquid, and distilling acetic acid and acetone from the liquid at a reduced pressure at a temperature under substantially 70° C., whereby to form a purified extract.

4. The method of concentrating vitamins B and G from rice polishings which comprises subjecting the material to the solvent action of a mixture of substantially 25% by volume of alcohol with water and containing about .5% of acetic acid, separating solid and liquid, distilling alcohol and water from the liquid under reduced pressure at a temperature under substantially 70° C., adding glacial acetic acid to the residue to dissolve the vitamin content thereof, diluting the mass with anhydrous acetone to form a mixed solvent in which the vitamin is soluble and in which an impurity is insoluble, separating the insoluble impurity from the liquid, distilling the liquid to remove acetic acid and acetone under reduced pressure at a temperature not over substantially 70° C., whereby to form an acidified residue containing the vitamins, adding water to the residue, and simultaneously volatilizing water and acid from the mass, whereby to form a stable slightly acidified vitamin extract.

In testimony whereof, I affix my signature.

BARNETT SURE.

UNITED STATES PATENT OFFICE.

HOMER T. YARYAN, OF TOLEDO, OHIO.

PROCESS FOR THE TREATMENT OF FLAXSEED, &c.

SPECIFICATION forming part of Letters Patent No. 241,772, dated May 17, 1881.

Application filed April 15, 1881. (No specimens.)

To all whom it may concern:

Be it known that I, HOMER T. YARYAN, of Toledo, Lucas county, Ohio, have invented a new and useful Improvement in Process for the Treatment of Flaxseed, &c., of which the following is a specification.

In the extraction of oil from flaxseed by the use of a liquid solvent and the subsequent removal of the solvent with superheated steam I have found that when the seed in its natural condition is crushed sufficiently to obtain the best results in yield of oil the meal is so fine as to prevent rapid percolation, and in the subsequent introduction of superheated steam its fineness prevents the penetration of steam with ease into the mass. To avoid these difficulties and to loosen the oil from its cells I dampen the seed before crushing with water or steam. I have found by experience that the best results are obtained by dampening the seed sufficiently to cause the grain to swell and burst the oil-cells, then to pass the seed through a drier to remove the excess of moisture, and at the same time toughen the hull.

The drying may be done after the crushing of seed, or only a sufficient amount of moisture may be added as not to require drying; but the best results are as described above.

I am aware that for many years water and steam have been added to flaxseed after crushing in the process of extracting oil by pressure; but this I do not claim. My process applies only where a hydrocarbon solvent is used for extracting the oil by percolation. An apparatus for this purpose is described in Patent No. 205,328, issued to me June 25, 1878.

I claim as new—

The process which consists in adding water or steam to flaxseed before crushing or grinding, in combination with any process for extracting oil by percolation with a hydrocarbon solvent, for the purpose of facilitating the process of extracting the oil.

HOMER T. YARYAN.

Witnesses:
E. O. BROWN,
F. B. DODGE.

In the United States Court of Appeals for the District of Columbia

APPEAL No. 8016

EZRA LEVIN, APPELLANT

v.

CONWAY P. COE, COMMISSIONER OF PATENTS, APPELLEE

APPEAL FROM THE JUDGMENT OF THE DISTRICT COURT OF THE UNITED STATES FOR THE DISTRICT OF COLUMBIA

BRIEF AND APPENDIX FOR APPELLEE

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In the United States Court of Appeals for the District of Columbia

APPEAL No. 8016

EZRA LEVIN, APPELLANT

v.

CONWAY P. COE, COMMISSIONER OF PATENTS, APPELLEE

APPEAL FROM THE JUDGMENT OF THE DISTRICT COURT OF THE UNITED STATES FOR THE DISTRICT OF COLUMBIA

BRIEF FOR THE COMMISSIONER OF PATENTS

This is an appeal from the judgment (Appellant's Appen. 11) of the District Court of the United States for the District of Columbia dismissing the Complaint (Appellant's Appen. 2) in which appellant sought to have the Court authorize the issuance of a patent containing claims 3, 4, and 6 to 16, inclusive, of appellant's application for patent, Serial No. 94,709, entitled "Method of Treating Cereal Grain Germ."

APPELLANT'S APPLICATION

Appellant's application (Appellant's Appendix 39–49) relates to a method of treating the germ portion of cereal grain and to the product obtained by the method. In his method appellant treats the germ of a cereal, as, for example, wheat germ, with a solvent such as ethlyene dichloride or carbon tetrachloride, for the purpose of extracting from the germ the oily constituent thereof. Appellant removes the oil because it has been found that oil is the cause of the rancidity which will develop in course of time if it is left in the germ. After the oil has been extracted appellant subjects the residual por-

tion of the germ, which contains the vitamin B complex, to treatment by steam for the purpose of removing from the residue all the solvent which remains therein. This last step is necessary because the solvent if left in the residue would make it unpalatable.

Some of the claims on appeal are for a product thus obtained and the remainder are for the method of treatment.

SUMMARY OF ARGUMENT

I. The steps of removing oil from cereal germs by solvent extraction and subsequently treating the residue to make a food product of high vitamin B content are disclosed in the Hoffman patent. It was not invention to remove the solvent from the oil-free residue obtained by Hoffman and omit the further treatment prescribed by him.

II. The Hoffman patent discloses carbon tetrachloride as a solvent which is suitable for extracting oil from cereal germs. This is one of the solvents described in appellant's application and with which he states he had great success. All of the claims on appeal, except one, are broad enough to cover the use of carbon tetrachloride as the solvent. The maintenance of an acid condition in the residue is inherent in the use of carbon tetrachloride as a solvent and the advantage of maintaining an acid condition to preserve the vitamin B content is shown by the prior art.

III. The use of steam to remove a solvent from a residue is conventional and this was admitted by appellant's witness, Wurster. It is also shown in the prior art.

IV. The features set forth in the claims on appeal are all found in the prior art.

V. Claims 7 and 13 are not in proper form. They are product claims in which the product is defined in part by a method step.

ARGUMENT

I

The patent to Hoffman, No. 1,640,193 (Appellant's Appen. 84) discloses a process in which cereal germs are freed from their vegetable oil by a suitable extracting agent, such as benzol

or carbon tetrachloride. The residue is then subjected to various operations, including cooking and malting for the purpose of gelatinizing and saccharifying the starch. As the examiner properly said in his statement (Appellant's Appen. 76)—

> Obviously, if this formation of soluble sugars is not desired, the steps and their corresponding functions could be omitted.

To obtain the product which appellant claims, it is only necessary to remove the solvent from the residue constituting the intermediate product obtained by Hoffman. The lower Court, in its Finding of Fact No. 8 (Appellant's Appen. 10) found that it was not invention to remove the solvent from the intermediate product of Hoffman by the method or process set forth in appellant's claims and omit the last stages of the Hoffman process.

Appellant admitted in his testimony (Appellant's Appen. 28) that the intermediate material or residue obtained by Hoffman may be assumed to have the same amount of vitamin B as that contained in appellant's product.

II

The solvent preferred by appellant for use in extracting the oil from the cereal germ is ethylene dichloride, but appellant also stated in his application (Appellant's Appen. 45) that excellent results have also been obtained with carbon tetrachloride as the solvent.

All of the claims on appeal, except one (claim 9) are broad enough to include the use of carbon tetrachloride as the solvent. Appellant contends that by using a suitable solvent the same will hydrolyze in conjunction with the moisture contained in the cereal germ to produce an acid, for example, hydrochloric acid, and that the acid condition thus produced is favorable to the preservation of vitamin B. It will be shown, however, that the fact that vitamin B is preserved by the presence of an acid condition was well known.

The patent to Sure, 1,869,721 (Appellant's Appen. 90), deals with the extraction of vitamins from various materials, such as rice polishings, wheat embryo, yeast, etc. Sure points out that

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vitamin B is less stable towards heat and in the presence of alkalies (Appellant's Appen. 90, lines 66 to 68). He further states (Appellant's Appen 91, lines 80 to 96) that in his process he uses some acid to stabilize the vitamin B, and that slight acidity is all that is required.

The publication "The Vitamins," Sherman and Smith, 1922 (Appellee's Appendix), states that—

> A few experiments on the stability toward acids and alkalies of water-soluble B in the form of an alcohol solution of wheat embryo were also reported. These tended to confirm similar tests of the antineuritic vitamin in its rapid destruction by alkalies and relatively marked stability toward hydrochloric acid.

Appellant admitted in his testimony (Appellant's Appen. 29) that if the Hoffman intermediate product were subjected to treatment by live steam the carbon tetrachloride would hydrolize somewhat to bring about an acid condition in the residue, which would preserve the vitamin B content.

Claim 9 specifies the use of ethylene dichloride but it is submitted that appellant is in no position to contend that this solvent is a patentable variant of carbon tetrachloride since appellant has treated these solvents in his application as equivalents.

III

Appellant sets forth in claims 11, 14, and 15 (Appellant's Appen. 4) the use of steam or heated moisture to remove the solvent from the residue after the oil has been extracted. That this method of removing a solvent from a product is not new is shown by the patent to Yaryan, 241,772, dated May 17, 1881 (Appellant's Appen. 95). In this patent reference is made in lines 7, 8, and 9 to the extraction of oil from flaxseed by use of a liquid solvent and the subsequent removal of the solvent with superheated steam.

Appellant's witness Wurster (Appellant's Appen. 33) stated on direct examination that it was common practice to subject solid materials to the action of steam, or steam in connection with vacuum, to remove the last traces of solvent there-

from. The same witness had been asked on direct examination the following very restricted question and answered in the negative (Appellant's Appen. 31):

> Q. Now, will you state, Mr. Wurster, whether in your work in the chemical engineering field you know of any instance in which an organic solid material has been treated with solvent and after the treatment with solvent the material has been then processed to the point where the residue is fit for human consumption?

It will be noted that this question is limited to organic material and solid material and to a residue fit for human consumption. The same witness admitted that the removal of a solvent from fibrous materials used for animal consumption had been done for a great many years and that it had also been done with vegetable oils suitable for animal consumption. Appellant himself in his testimony (Appellant's Appen. 24) said that he had thoroughly searched the literature for ten years and had never found a single substance, except coffee, which had been treated with an organic solvent or solvent extraction leaving a residue fit for human food. Appellant also stated that coffee is not a food.

It thus appears that appellant admits that solvent extraction has been used on various organic materials, fibers, and liquid, to produce a food product suitable for animal consumption, and that it has also been used on a material, coffee, for human consumption. Yet appellant urges that the same method of treatment of analogous material for use as a food for human beings amounts to invention. But, as the Supreme Court said in *Paramount Publix Corp.* v. *American Tri-Ergon Corp.*, 294 U. S. 464:

> The application of an old process to a new and closely analogous subject matter, plainly indicated by the prior art as an appropriate subject of the process, is not invention.

See also *In re Williams*, 87 F. (2d) 499, 24 C. C. P. A. 861; *In re Migrdichian*, 108 F. (2d) 237, 27 C. C. P. A. 806; *In re Foster*, 86 F. (2d) 981, 24 C. C. P. A. 794.

IV

Claims 3, 6, 7, 12, 13, and 16 (Appellant's Appen. 3 to 5) recite the product resulting from appellant's process. All of these claims, it is submitted, are met by the intermediate product produced by Hoffman if that product is treated by conventional steps for the removal of the solvent remaining therein. This was found by the lower court not to involve invention. See Finding of Fact No. 8 (Appellant's Appen. 10.)

The remaining claims 4, 8, 9, 10, 11, 14, and 15 (Appellant's Appen. 3 and 4) recite the method employed by appellant for producing the product. The first step of the method—that is, the removal of the oil by means of a solvent—is clearly disclosed by Hoffman. As was pointed out above, the production of an acid condition is inherent in the Hoffman method because the solvent disclosed by him, carbon tetrachloride, hydrolizes with the moisture in the cereal grain and thus produces the acid condition. The use of steam or heated moisture to drive off the solvent from a residue is admittedly old (Appellant's Appen. 33, 34) and is also shown in the Yaryan patent.

Method claim 8 recites that the solvent is removed from the germ under sub-atmospheric pressure and at a temperature of the order of 125° to 160° F. The sub-atmospheric pressure is used in order that the solvent may be driven off at a relatively low temperature so that the vitamin B will not be injured. This is not new, however, for the Sure patent clearly describes (Appellant's Appen. 92, beginning line 1) the use of a partial vacuum and temperature between 50° C. and 60° C., which are equivalent to 122° to 140° F. in the treatment of material containing vitamin B. The purpose stated is to preserve the vitamin B.

V

Claims 7 and 13 (Appellant's Appen. 3 and 4) are for a product. They are not limited to the recitation of the product but include the process limitation that the germ is freed from the oil by solvent extraction. Obviously a process

limitation has no place in a product claim. The claims were held unpatentable for this reason by the Examiner, Board of Appeals, and by the lower Court. It is submitted that this holding was in accordance with the principle set forth by this Court in *In re Jones,* 58 App. D. C. 379, 30 F. (2d) 1003.

It is to be noted that appellant's assignment of errors contains no reference to this rejection, nor is any reference made to it in appellant's brief.

CONCLUSION

It is submitted that the lower Court properly held that none of the claims on appeal is patentable and that its judgment should be affirmed.

W. W. COCHRAN,
Solicitor, U. S. Patent Office,
Attorney for Appellee.

NOVEMBER 1941.

APPELLEE'S APPENDIX

Extract from the publication "The Vitamins," by Sherman and Smith, 1922:

> A further study of the solubility and stability of the water-soluble growth-promoting vitamin was made by McCollum and Simmonds (1918) using white beans or wheat embryo as sources of the vitamin. The technique employed in this and other similar studies
> 307 consisted in feeding young rats a diet of purified food substances (casein, salt mixture, agar-agar and dextrine) together with 5 percent of butterfat to supply an abundance of vitamin A. The rats were confined to this diet for about five weeks until they had either become stationary in weight or were declining with evidences of paralysis. The material to be tested for vitamin B was then added to the diet, the animals continuing to decline or responding with growth, according as the material was deficient or rich in this vitamin. This method was considered to show within two weeks whether the preparation under investigation contained vitamin B in significant amounts. As tested by these criteria, it was shown that vitamin B is not extracted directly from beans, wheatgerm, or pig kidney by ether, benzene, or acetone, but is readily extracted in great part by alcohol (95 percent). After being removed by alcohol it was shown to be soluble in benzene but very slightly soluble in acetone. McCollum and Simmonds conclude that: "The probability that there should be two or more physiologically indispensable substances in what we term water-soluble B, both or all of which should show the same solution relations with three solvents, is relatively small, and lends support to our view that the substance which

protects animals polyneuritis is the only essential complex in the extracts described." A few experiments on the stability toward acids and alkalies of water-soluble B in the form of an alcohol solution of wheat embryo were also reported. These tended to confirm similar tests of the antineuritic vitamin in its rapid destruction by alkalies and relatively marked stability toward hydrochloric acid. Nitrous acid had slight if any action, this fact constituting a strong indication that the active substance is neither a primary nor a secondary amine.

U. S. GOVERNMENT PRINTING OFFICE: 1941

APPELLANT'S REPLY BRIEF

IN THE

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA,

UNITED STATES
COURT OF APPEALS FOR
DISTRICT OF COLUMBI

FILED DEC 31 1941

Joseph W. Stewart

No. 8016

EZRA LEVIN, c/o BIOVIN CORPORATION, 155 NORTH CLARK STREET, CHICAGO, ILLINOIS,
Plaintiff, Appellant,

vs.

CONWAY P. COE, COMMISSIONER OF PATENTS,
Defendant, Appellee.

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THE GUNTHORP-WARREN PRINTING COMPANY, 210 WEST JACKSON, CHICAGO

IN THE

United States Court of Appeals

FOR THE DISTRICT OF COLUMBIA,

No. 8016

EZRA LEVIN, c/o BIOVIN CORPORATION, 155 NORTH CLARK STREET, CHICAGO, ILLINOIS,

Plaintiff, Appellant,

vs.

CONWAY P. COE, COMMISSIONER OF PATENTS,

Defendant, Appellee.

APPELLANT'S REPLY BRIEF.

Appellee's argument is that steam and vacuum had been used to extract solvent from all kinds of material and that merely to apply the same processes to the removal of material from *food* is not invention.

We agree with that statement. It has no relationship to the facts in issue.

In the first place it is very easy to remove enough solvent from a fibrous material so that it will be fitted for non-consumption purposes. It is the removal of the last traces of solvent which is necessary to

fit the material for food purposes. The mere use of steam and vacuum is *not* sufficient to do this. Appellee's brief gives the impression that the various solvent extracted substances listed on pages 4 and 5 of the brief could have been used for food but merely did not happen to be so used. The fact is that they were unfit for use as food because of the presences of traces of solvent. This was made very clear by the testimony of Mr. Wurster at page 31 of the appendix. Applicant's process is the only one which will remove the last traces of solvent. These traces do not matter in products which are not eaten, and there were many processes which could remove enough solvent for non-sumptuary purposes. But these processes were not such as to produce a satisfactory food product. Applicant is the only one who has ever made use of the hydrolysis of a solvent, in combination with steam and vacuum, to remove the last traces of solvent and render a solid cellular material fit for eating after it has been treated with a non-palatable solvent.

Moreover, hydrolysis has not been used in this manner in connection with any product, food or otherwise, to remove last traces of solvent.

Appellee's brief refers to the use of carbon tetrachloride in Hoffman as if Hoffman used that material for hydrolysis purposes. He did not. Hoffman did not even attempt to remove solvent, obviously because he considered it impossible. His preferred solvent was benzene. Benzene does not hydrolyze. Obviously, if his less preferred solvent could not act to produce Levin's results, Hoffman did not expect any better result from his other solvent.

CONCLUSION.

I.

The steps of applicant's process are entirely new. They are not found in combination in the references, nor is the use of hydrolysis, to remove last traces of solvent, found separately.

II.

Applicant's process is the only one which ever did remove the last traces of solvent from any solid cellular material under conditions which would permit its direct use as a food. His process is the only one which will do this.

Respectfully submitted,

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